U.S. DEPARTMENT OF COMMERCE
National Technical Information Service
PB-290 106

Sampling System Evaluation for High-temperature, High-pressure Processes

Acurex Corp, Mountain View, CA Energy and Environmental Div

Prepared for

Industrial Environmental Research Lab, Research Triangle Park, NC

Aug 78

PB 290106

United States
Environmental Protection
Agency

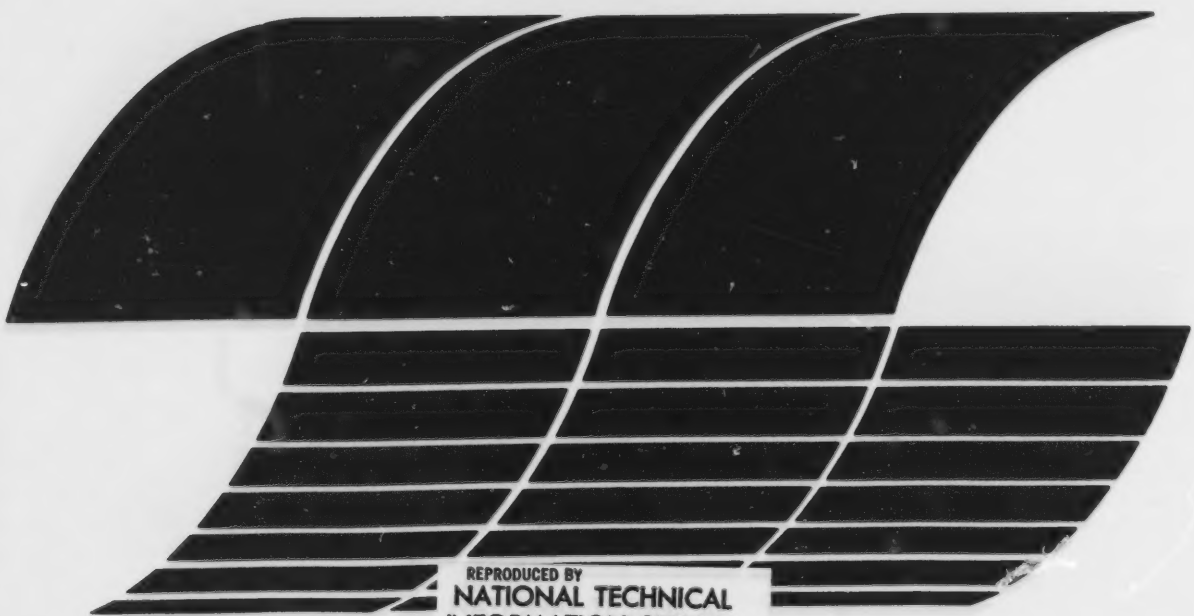
Industrial Environmental Research
Laboratory
Research Triangle Park NC 27711

EPA-600/7-78-158
August 1978

EPA

Sampling System Evaluation for High-temperature, High-pressure Processes

Interagency Energy/Environment R&D Program Report



REPRODUCED BY
NATIONAL TECHNICAL
INFORMATION SERVICE
U. S. DEPARTMENT OF COMMERCE
SPRINGFIELD, VA. 22161

113

RESEARCH REPORTING SERIES

Research reports of the Office of Research and Development, U.S. Environmental Protection Agency, have been grouped into nine series. These nine broad categories were established to facilitate further development and application of environmental technology. Elimination of traditional grouping was consciously planned to foster technology transfer and a maximum interface in related fields. The nine series are:

1. Environmental Health Effects Research
2. Environmental Protection Technology
3. Ecological Research
4. Environmental Monitoring
5. Socioeconomic Environmental Studies
6. Scientific and Technical Assessment Reports (STAR)
7. Interagency Energy-Environment Research and Development
8. "Special" Reports
9. Miscellaneous Reports

This report has been assigned to the INTERAGENCY ENERGY-ENVIRONMENT RESEARCH AND DEVELOPMENT series. Reports in this series result from the effort funded under the 17-agency Federal Energy/Environment Research and Development Program. These studies relate to EPA's mission to protect the public health and welfare from adverse effects of pollutants associated with energy systems. The goal of the Program is to assure the rapid development of domestic energy supplies in an environmentally-compatible manner by providing the necessary environmental data and control technology. Investigations include analyses of the transport of energy-related pollutants and their health and ecological effects; assessments of, and development of, control technologies for energy systems; and integrated assessments of a wide range of energy-related environmental issues.

REVIEW NOTICE

This report has been reviewed by the participating Federal Agencies, and approved for publication. Approval does not signify that the contents necessarily reflect the views and policies of the Government, nor does mention of trade names or commercial products constitute endorsement or recommendation for use.

This document is available to the public through the National Technical Information Service, Springfield, Virginia 22161.

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1. REPORT NO. EPA-600/7-78-158	2.
3. RECIPIENT'S ACCESSION NO. PB290106	
4. TITLE AND SUBTITLE Sampling System Evaluation for High-temperature, High-pressure Processes	5. REPORT DATE August 1978
	6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S) William Masters, Robert Larkin, Larry Cooper, and Craig Fong	8. PERFORMING ORGANIZATION REPORT NO.
9. PERFORMING ORGANIZATION NAME AND ADDRESS Acurex Corporation/Energy and Environmental Division 485 Clyde Avenue Mountain View, California 94042	10. PROGRAM ELEMENT NO. EHE623 and 624
	11. CONTRACT/GRANT NO. 68-02-2153
12. SPONSORING AGENCY NAME AND ADDRESS EPA, Office of Research and Development Industrial Environmental Research Laboratory Research Triangle Park, NC 27711	13. TYPE OF REPORT AND PERIOD COVERED Final; 3/76-7/77
	14. SPONSORING AGENCY CODE EPA/600/13

15. SUPPLEMENTARY NOTES IERL-RTP project officer is William B. Kuykendal, Mail Drop 62, 919/541-2557.

16. ABSTRACT The report describes a sampling system designed for the high temperatures and high pressures found in pressurized fluidized-bed combustors (PFBC). The system uses an extractive sampling approach, withdrawing samples from the process stream for complete analysis of particulate size, mass concentration, shape, and chemical composition. Two series of tests were run at Exxon's pilot-scale PFBC in Linden, New Jersey: the first was run to measure particulate mass and size distribution (particulate sizing was achieved using a commercial cascade impactor operating at about 105 C); the second was run to determine if condensation occurred between the process temperature of 720 C and the impactor temperature. Results show that cascade impactors can be successfully used for sizing in the high-temperature, high-pressure process stream of the PFBC, and that condensation was not a problem in the tests conducted.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS		b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Pollution	High Pressure Tests	Pollution Control	13B
Sampling	Fluidized Bed Processors	Stationary Sources	14B
Extraction	Analyzing	Particulate	07A/13H 13I
Evaluation	Dust	Mass Concentration	
High Temperature Tests	Impactors	Cascade Impactors	11G
Condensing			07D

18. DISTRIBUTION STATEMENT Unlimited	19. SECURITY CLASS (This Report) Unclassified	21. NO. OF PAGES 113
	20. SECURITY CLASS (This page) Unclassified	22. PRICE PC A06/MF A01

EPA Form 2220-1 (9-73)

EPA-600/7-78-158
August 1978

Sampling System Evaluation for High-temperature, High-pressure Processes

by

William Masters, Robert Larkin, Larry Cooper, and Craig Fong

Acurex Corporation/Energy and Environmental Division
485 Clyde Avenue
Mountain View, California 94042

Contract No. 68-02-2153
Program Element Nos. EHE623 and 624

EPA Project Officer: William B. Kuykendal

Industrial Environmental Research Laboratory
Office of Energy, Minerals, and Industry
Research Triangle Park, NC 27711

Prepared for

U.S. ENVIRONMENTAL PROTECTION AGENCY
Office of Research and Development
Washington, DC 20460

TABLE OF CONTENTS

Section		Page
1.	INTRODUCTION	1
2.	OVERVIEW OF RESULTS	3
	2.1 Phase I -- Demonstration Tests	3
	2.2 Phase II -- Condensation Tests	4
3.	EQUIPMENT DESCRIPTION	7
	3.1 Sampling System	7
	3.1.1 Phase I -- Demonstration Tests	7
	3.1.2 Phase II -- Condensation Tests	44
	3.2 Exxon Miniplant Test Facility	53
4.	TEST DESCRIPTION	60
	4.1 Phase I -- Demonstration Tests	60
	4.1.1 Procedures	60
	4.1.2 Pretest Activities	61
	4.1.3 First Run	61
	4.1.4 Second Run	62
	4.1.5 Third Run	62
	4.1.6 Post-Test Activities	63
	4.2 Phase II -- Condensation Tests	63
	4.2.1 Procedures	63
	4.2.2 Pretest Activities	63
	4.2.3 Runs 1 and 2	64
	Runs 3 and 4	65
	4.2.5 Post-Test Activities	65
5.	DATA AND RESULTS	66
	5.1 Phase I -- Demonstration Test Data	66
	5.1.1 Test Conditions	66
	5.1.2 Instrument Readings	66
	5.2 Phase I -- Demonstration Test Results	73
	5.3 Phase II -- Condensation Test Data	78
	5.3.1 Test Conditions	78
	5.3.2 Instrument Readings	98

Preceding page blank

TABLE OF CONTENTS (Concluded)

Section		Page
	5.4 Phase II -- Condensation Test Results	102
	5.4.1 General Results and Observations	104
	5.4.2 Initial Analyses for Condensation Effects . . .	111
	5.4.3 Alternate Approach	122
6	CONCLUSIONS	126
	REFERENCES	128

LIST OF ILLUSTRATIONS

Figure		Page
3-1	High-temperature, high-pressure sampling system.	11
3-2	System schematic	13
3-3	Exploded view of HTHP probe.	15
3-4	Aerotherm HTHP sampling probe and duct interface valve.	19
3-5	Access valves	21
3-6	HTHP probe housing/side view	23
3-7	HTHP probe housing/end view	24
3-8	HTHP probe/side view/front half	25
3-9	HTHP probe/side view/back half	27
3-10	HTHP probe rear access plate and controls/side and end view	28
3-11	HTHP probe rear access plate and controls/top view . . .	29
3-12	Dowtherm system.	30
3-13	Dowtherm console	31
3-14	Gas sampler train - flow control oven.	34
3-15	Oven assembly	35
3-16	Organic cartridge.	37
3-17	Organic module	38
3-18	Organic module -- exploded view	39
3-19	Flow control oven and gas train.	41
3-20	Organic module and impinger bottles.	43
3-21	Control consoles	45
3-22	Probe configurations	47
3-23	Scalping cyclone assembled	49

LIST OF ILLUSTRATIONS (Continued)

Figure		Page
3-24	Scalping cyclone disassembled.	50
3-25	Transport tube	52
3-26	Pressurized fluidized bed coal combustor system	54
3-27	HTHP probe assembly installed at Exxon miniplant	57
5-1	Probe inlet bias	72
5-2	Particle size distribution	76
5-3	Impactor substrates	79
5-4	Impactor substrates	81
5-5	Impactor substrate -- Run 3, Stage 5	83
5-6	Impactor substrate -- Run 2, Stage 5	85
5-7	Particle photomicrographs Stage 1	87
5-8	Particle photomicrographs Stage 2	89
5-9	Particle photomicrographs Stage 4	91
5-10	Particle photomicrographs Stage 6	93
5-11	Particle chemical composition	95
5-12	Condensation of H_2SO_4	101
5-13	Particle Photomicrographs -- Run 1, front filter	107
5-14	Particle photomicrographs -- Run 4, front filter	109
5-15	Particle photomicrographs -- Run 4, rear filter	113
5-16	Particle photomicrographs -- Run 1, rear filter	115
5-17	Particle chemical composition -- blank rear filter . . .	117
5-18	Particle chemical composition -- Run 4	119

LIST OF TABLES

Table		Page
3-1	Sampling System Capabilities	9
3-2	Utility Requirements	59
5-1	Test Conditions	67
5-2	Phase I Probe Instrumentation Readings	68
5-3	Gas Train Instrument Readings	69
5-4	Anisokinetic Correction Factors -- Phase I	70
5-5	Structure Temperatures	74
5-6	Particulate Content	75
5-7	Particle Size Distribution	77
5-8	Exxon -- Phase II FBC Operating Conditions	97
5-9	Probe Instrumentation Readings	99
5-10	Anisokinetic Correction Factors -- Phase II	103
5-11	Particulate Concentration	105
5-12	Visual Observation of Filters -- Phase II	106
5-13	Concentration of Elements in Flyash SSMS Analysis (Partial)	123
5-14	Partial Comparison of Front and Rear Particulate Catches from Exxon Test Series I and II	124

SECTION 1

INTRODUCTION

Advanced coal conversion processes present new problems in particulate sampling, including severe environments beyond the capabilities of conventional equipment.

Fluidized bed combustion and coal gasification processes emit gases containing large quantities of fine particles. These particles must be removed to prevent damage to process equipment (mainly turbines) and to eliminate potential environmental pollution. Development of particulate removal equipment is an important step toward making advanced coal conversion processes practical. The sampling system described in this report is one of the first tools available for measuring the collection efficiency of fine particle removal devices operating in high-pressure, high-temperature environments. The sampling system described in this report is specifically designed for the high temperatures and pressures found in pressurized fluidized bed combustors (PFBC). The system uses an extractive sampling approach, withdrawing samples from the process stream for complete analysis of particulate concentration, shape, size, and chemical composition. The capabilities of the new system have been demonstrated in two phases of sampling operations at Exxon Corporations pilot-scale pressurized, fluidized bed combustion Miniplant located in Linden, New Jersey. The first phase of testing was performed

during the week of March 21, 1977 with the sample probe in its basic configuration. The second test phase used modified probe internals designed to investigate possible condensation of alkali metals as a result of cooling the sample prior to collection. These tests were performed the week of May 23, 1977. The system performed successfully in a variety of operating modes, producing sample data from both test series.

Acurex has developed the HTHP sampling system for the Industrial Environmental Research Laboratory of the U.S. Environmental Protection Agency. This work is part of the program, Measurements of High-Temperature, High-Pressure Processes (Contract 68-02-2153), intended to produce the new sampling technology needed for advanced coal conversion processes. The EPA project officer was William B. Kuykendal.

An overview of the results of both sampling operations is presented in Section 2. Section 3 discusses the sampling system equipment in detail and gives a brief description of the Exxon Miniplant test facility. Section 4 describes assembly and operating procedures and contains a narrative of events in each phase of tests. All results and supporting data are presented in Section 5 while Section 6 summarizes the program conclusions.

SECTION 2

OVERVIEW OF RESULTS

This Section presents an overview of the results from both series of sampling operations, including the Phase I demonstration tests and the Phase II condensation tests. A more thorough discussion of the results can be found in Section 5.

2.1 PHASE I -- DEMONSTRATION TESTS

The objective of the first sampling operation was to demonstrate the HTHP sampling system's range of capabilities. Three sampling runs were successfully made: one using a filter* to collect total particulate, and two using a cascade impactor to collect particle sizing data. Trace metals and trace organics sampling equipment was operated during the filter run. The tests produced the following data:

- Particulate concentration
- Particulate size distribution
- Moisture content
- Particulate chemical composition
- Particulate shape

*A complete detailed description of the sampling equipment is given in Section 3.

- Duct gas temperature and pressure
- Access port and valve external surface temperatures

The test also produced samples of trace organics collected on XAD-2 porous polymer sorbent and trace elements collected in oxidizing impinger solutions. However, the analysis of these samples is beyond the scope of the HTHP program.

The test series demonstrated the capability of the sampling system to operate in a severe PFBC environment of 740°C (1360°F) and 910 kPa (9 atm). Generally, the system operated as designed with regard to: obtaining access to the pressurized duct while the process was operating, inserting the sampling probe, sampling the gas stream, and withdrawing the sampling probe. However, as might be expected in a first field test demonstration, a few hardware problems were found. Most of these were corrected before the sampling tests, but one uncorrected problem, a malfunctioning impactor heater, gave sample collection temperatures which were lower than desired. The heater was replaced for the second phase of testing.

During the test sequence, the sampling operations proceeded very smoothly. The three sampling runs were completed within a 30-hour period (20 working hours). The tests showed the versatility of the system, operating with two different types of particle collectors, with and without trace element sampling equipment.

2.2 PHASE II -- CONDENSATION TESTS

The goal of Phase II was to determine the effect of sample cooling on measured particlate mass and composition. We were particularly concerned that trace elements might condense in the probe in significant quantities between process temperature and 450°F. The condensation

effects tests were also successfully performed. A total of four separate runs were completed with the cyclone-dual filter configuration. The only difference between the runs was the sampling time of each, and, in the case of test number three and four, two Saffil alumina filters were "sandwiched" to comprise the front filter (at stream conditions). Tests 1 and 2 used a single front filter. All other conditions, both in the FBC system and in the sampling system remained, nominally, the same. Trace element and trace organic sampling equipment were not employed in the Phase II tests.

Again, as in the first test series at the Miniplant, certain hardware problems prevented some measurements, the most important of which, was the malfunctioning of the stack thermocouple. This temperature is approximated at 730^{o}C (1350^{o}F), the average stack temperature during the first test series. Very similar PFBC system operating conditions between the two test series make this a reasonable approximation. Another less critical malfunction occurred in the transport tube outlet filter thermocouple. This problem was an intermittent electrical short. Consequently, the measurement could be made only periodically. Another problem occurred with the scalping cyclone. Its protective gold plating blistered and peeled causing gold contamination in the cyclone and front filter catches. The resultant oxidation of the titanium cyclone body did not appear to contribute to the contamination problem.

As previously mentioned, the purpose of the Phase II tests was to investigate the effect of sample cooling on measured particulate mass and composition. Based on one test at one set of PFBC operating conditions, the Phase II tests showed no apparent indication of trace element

condensation at the reduced sample collection temperature. In other terms, we can say that sample cooling seems to have had little effect on measured particulate mass and composition.

SECTION 3

EQUIPMENT DESCRIPTION

The following discussion is divided into two parts: (1) a description of the advanced sampling system, this section being further divided to discuss the original probe configuration and the modified configuration for the condensation effects tests, and (2) a description of the PFBC Miniplant test facility.

3.1 SAMPLING SYSTEM

The sampling system described in this report samples particulate, trace organics and trace metal contaminants in high-pressure, high-temperature gas streams. The system represents an advancement in the state of the art designed to sample new coal conversion processes. This section is divided into a discussion of the sampling as it was used both in Phase I and Phase II tests. Since there were many similarities between the two systems, such as the pressure containment vessel and traversing mechanism, only the differences between the two probe configurations will be mentioned in Section 3.1.2.

3.1.1 Phase I -- Demonstration Tests

The basic functions of the sampling system are to:

- Safely contain facility pressure
- Insert the sample probe into the process duct while the process is operating
- Extract a representative sample

- Cool the sample to a temperature which is compatible with developed particle collectors yet prevents condensation ($230^{o}C$)
- Collect and aerodynamically size particulates
- Collect trace organics and trace metals
- Monitor duct conditions and control sample flowrate to give accurate isokinetic capture conditions
- Remove the sample probe and close off duct access so that collected samples may be removed while the process remains pressurized

To perform these functions, the sampling system includes the following subsystems:

- Sample probe assembly
- Dowtherm coolant system
- Hydraulics for probe traverse actuation
- Flow control oven
- Trace organics module
- Trace metal impinger train
- Control consoles

System capabilities are described in Table 3-1. Figures 3-1 and 3-2 show system schematics of the HTHP sampler.

3.1.1.1 Probe Assembly

The probe assembly includes the sample probe, probe housings, and duct access valves. The sample probe itself, shown in Figure 3-3, consists of the sample nozzle, pitot tube, Dowtherm-cooled section with manifold, a sample particulate collection device, a flowmeter, a heated transport tube section, transducer and control devices mounted on the

TABLE 3-1. SAMPLING SYSTEM CAPABILITIES

Sample Environment	
• Temperature	650°C - 1000°C (1200°F - 1800°F)
• Pressure	300 - 2000 kPa (3 - 20 atm)
• Gas Constituency CO CO_2 NO SO_2 H_2O NO_x H_2S trace organics trace inorganics	Concentrations subject to further investigation, dependent on process sampled
• Stream Velocity	2 - 46 m/s (8 - 150 fps)
• Particulate Grain Loadings	0 - 34.3 g/m^3 (0 - 15 gr/ft^3) (subject to further consideration and actual process characteristics)
• Particulate Size Range (for classification)	0.2 - 26 microns (Notes: Larger particulates may be acceptable in most cases of total mass determination, or if classified, they may be amenable to "scalping" ahead of classification device)
• Duct Size	Variable depending on probe; std. is 20.3 cm (8 in.) I.D. minimum
Sampling System Configuration	Modular, so as to allow in-situ or extractive sampling by cooled probe
Traverse Capability or Penetration of Nozzle into Duct or Vessel	Approximately 46 cm (18 in.) either in-situ or extractive configuration (some dependence on internal configuration of duct or vessel). Can be extended by relatively minor hardware modification (longer probe, chamber extension, spool piece, etc.)

TABLE 3-1. (Concluded)

Access Process Port Requirements	Standard: 10.2 cm (4 in.) IPS minimum, 136 kg (300 lb.) flange access through 10.2 cm (4 in.) IPS alloy gate value (Note: Smaller ports may be acceptable if special probe assembly is used)
Stream Constituency Analysis	Particulates, gases (inorganic and organic, trace elements, trace organics)

S-038b

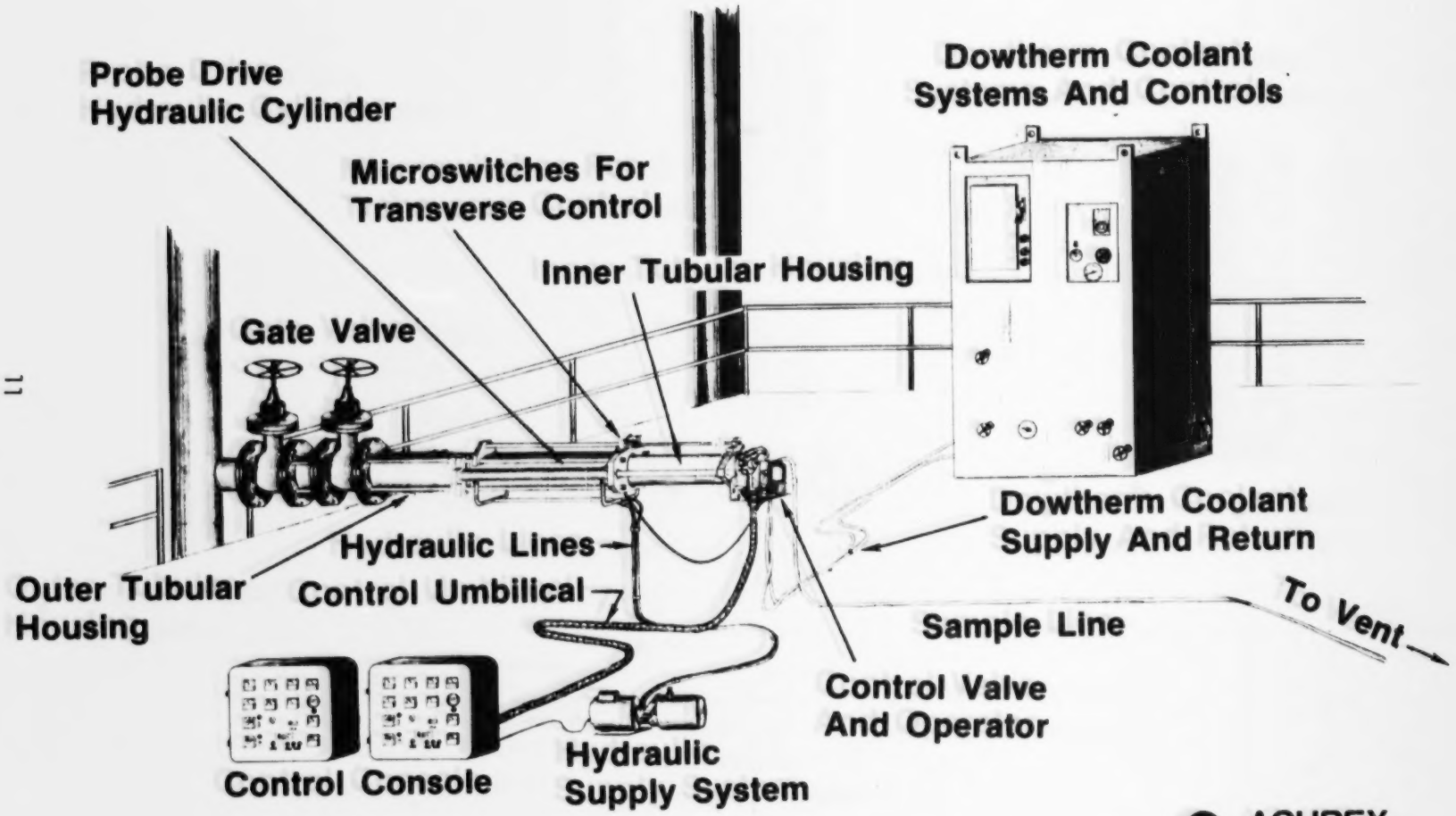


Figure 3-1. High-temperature, high-pressure sampling system.

ACUREX
Aerotherm

Preceding page blank

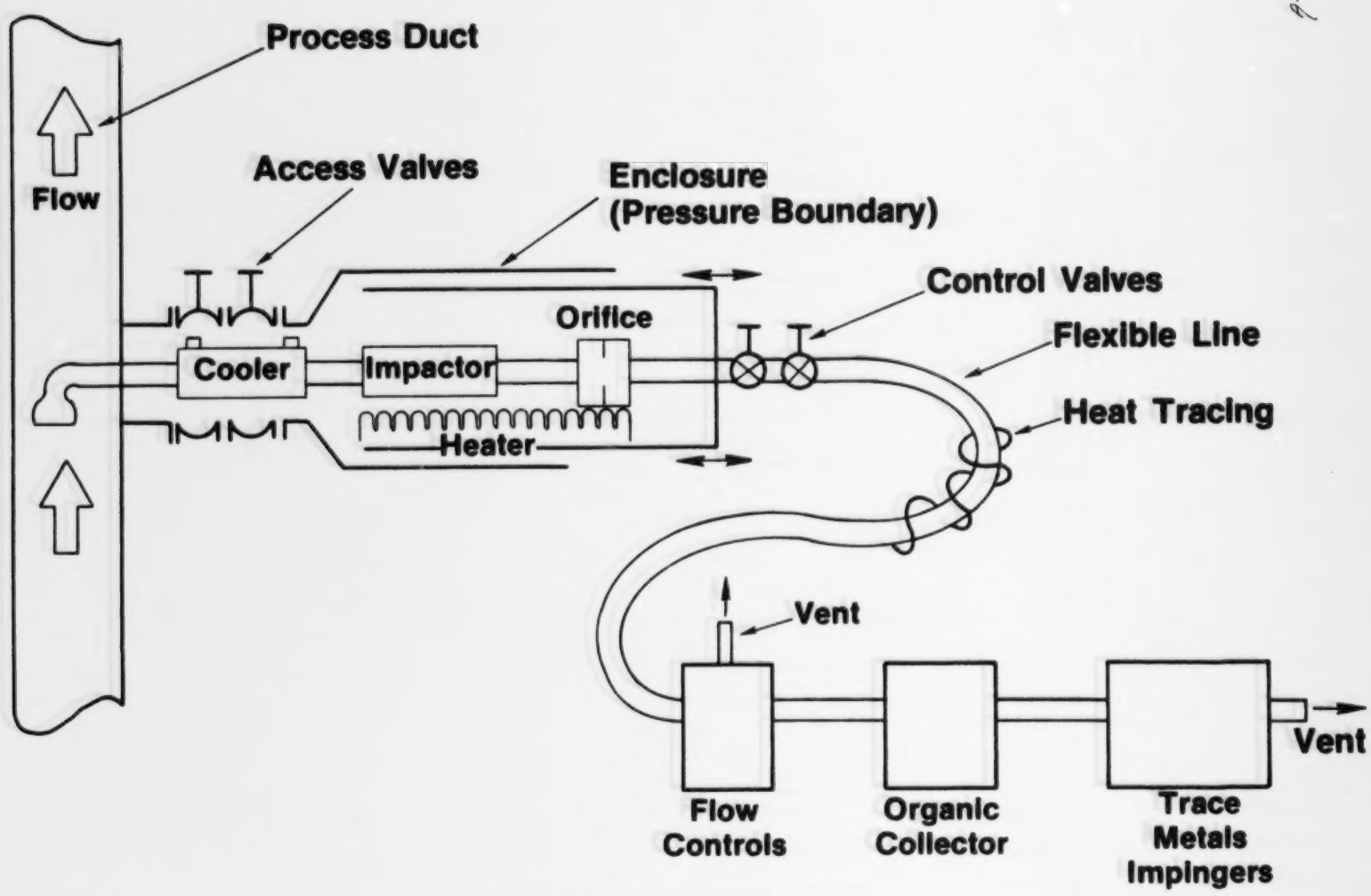


Figure 3-2. System schematic.

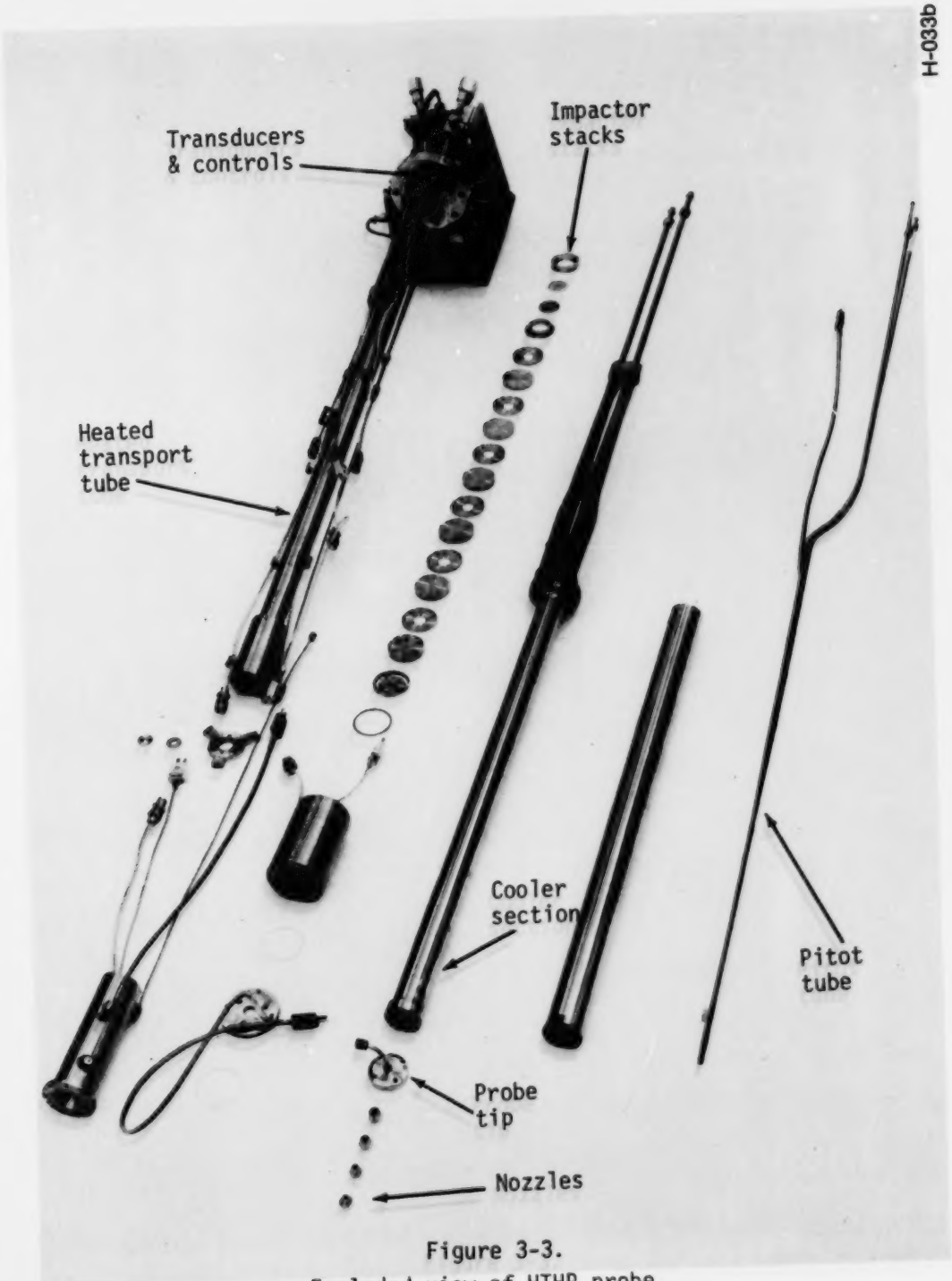


Figure 3-3.
Exploded view of HTHP probe.

Preceding page blank

end of the probe. All probe components, internal and external, that would be exposed to temperatures in excess of 1000°F, were constructed of 625 Inconel. These include the probe tip, nozzle, cooling section, and transport tube up to the cascade impactor. Most other probe components were constructed of grade 316 stainless steel. The assembled sample probe is then installed in an inner probe housing. The inner probe housing is a tube that telescopes into an outer probe housing and is sealed by a bolted flange and sliding seal. The outer probe housing, on its laboratory support stand designed to a factor of safety of 4.0, is shown in Figure 3-4. The outer probe housing is bolted to the process duct by two 10.2 cm (4-in) diameter gate valves in series. This 10.2 cm (4-in) opening provides clearance for the sample probe to be inserted into the sample stream, as shown in Figure 3-5. The entire telescoping system, shown in Figures 3-6 and 3-7, is driven by hydraulic cylinders.

Two types of sample particulate collection devices were used in the demonstration tests. They were a University of Washington Mark III cascade impactor with seven stages of particulate sizing and a glass fiber thimble filter with a large total mass capacity but with no sizing capability.

The inlet nozzle is interchangeable to allow isokinetic sampling. The nozzle diameter used was 1.9 cm (0.75 in) for all tests and was sized according to the anticipated Exxon miniplant stream conditions. The actual stream velocity (a calculated, not measured value) was not as close to expected conditions as would be desirable. A discussion of isokinetic sampling rates is in Section 5.

Preceding page blank

H-0356

Figure 3-4.
Aerotherm HTHP sampling probe and duct interface valve.

Preceding page blank

H-0536

Figure 3-5.
Access valves.

Preceding page blank

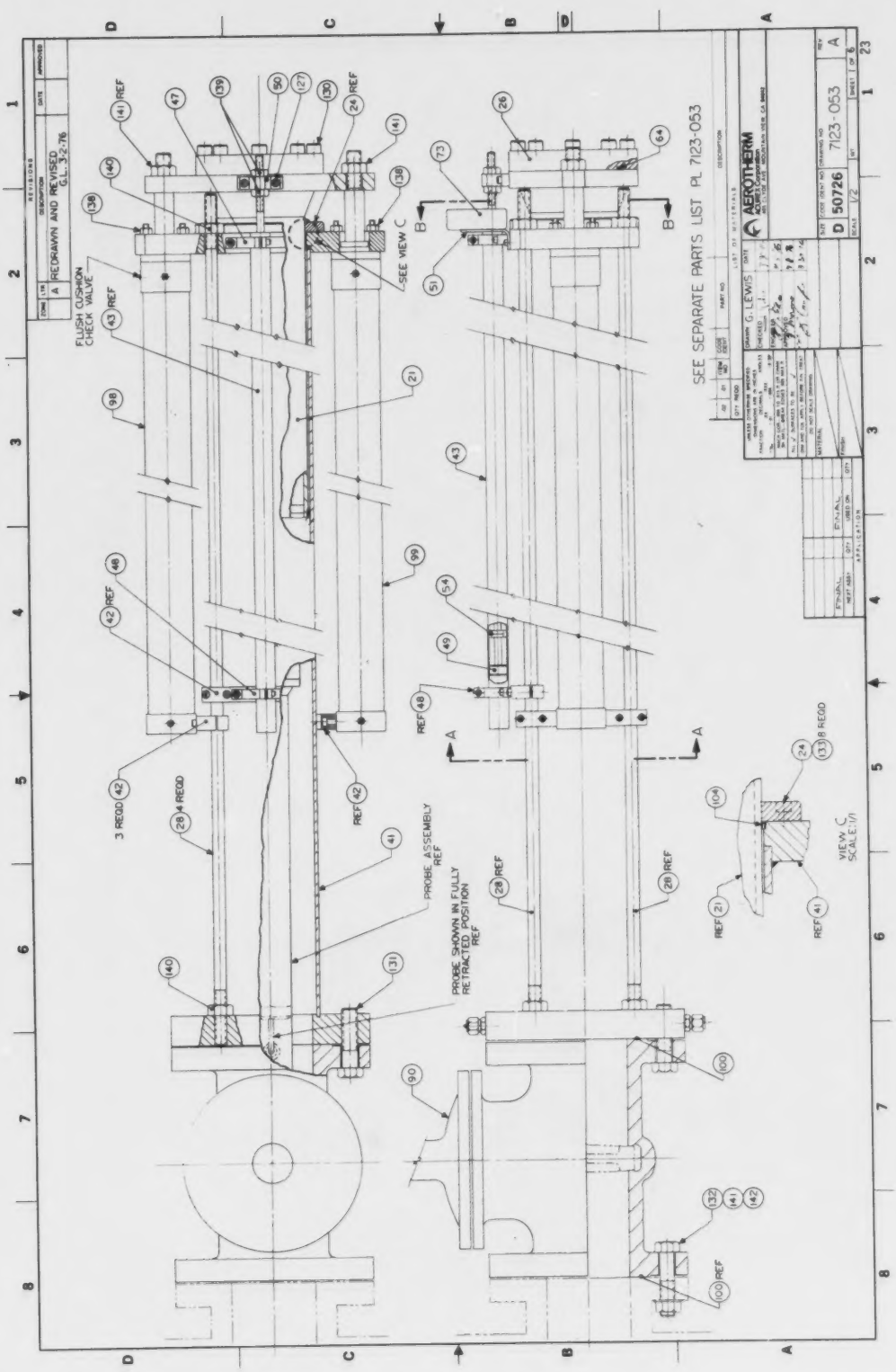


Figure 3-6. HTHP probe housing/side view.

Preceding page blank

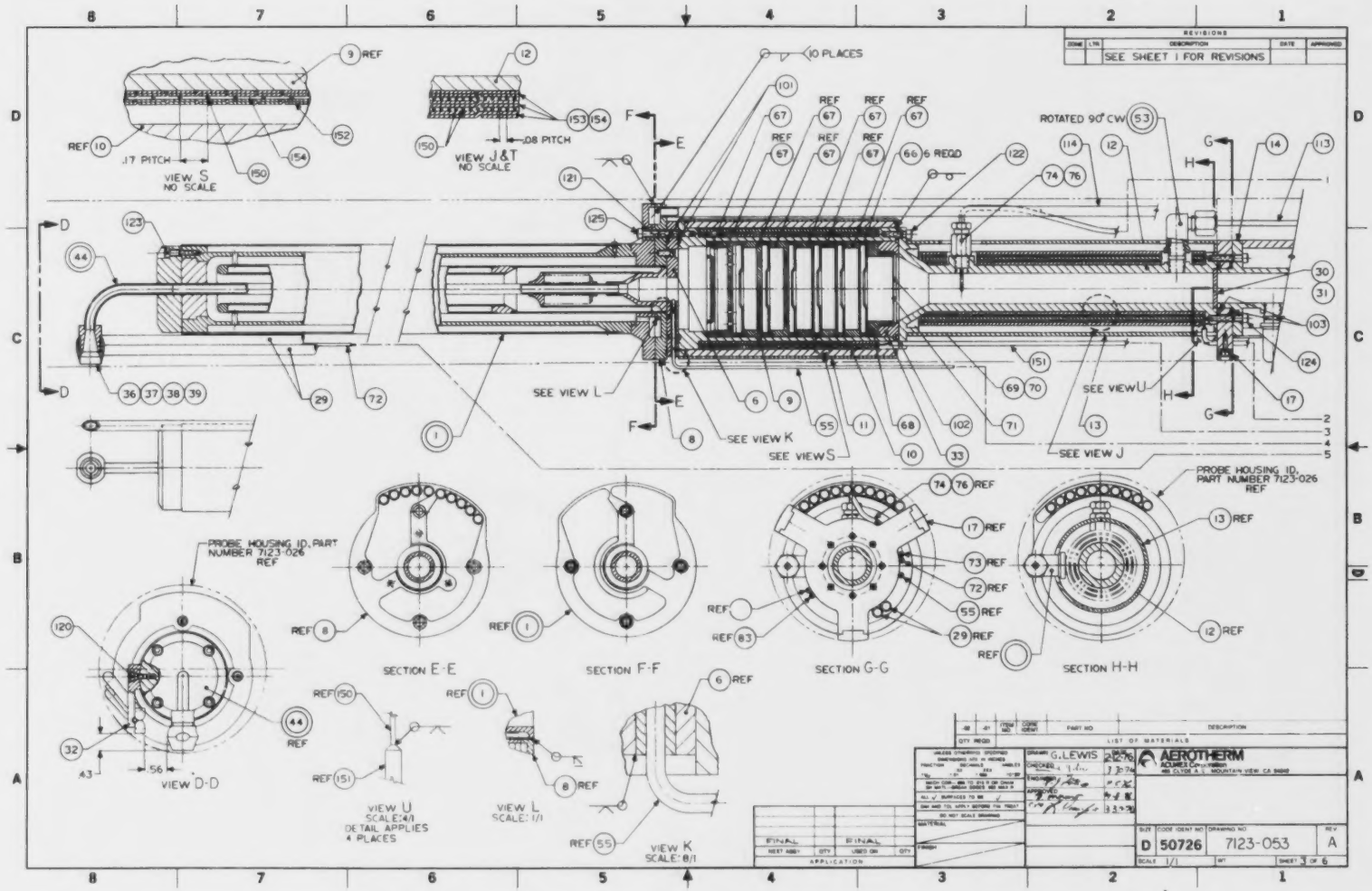


Figure 3-8. HTHP probe/side view/front half.

Sample gas enters the inlet nozzle isokinetically and travels through the Dowtherm-cooled section, shown in Figure 3-8. The sample gas then passes through one of the particulate collection devices mentioned above. It then goes to a flowmeter and an electrically heated and controlled transport tube section which is mounted to and routed through a rear access plate. Process pressure, velocity pressure, orifice differential pressure, and sample gas temperature instrumentation lines, lead aft to the access plate, as shown in Figures 3-8 and 3-9. Thermocouples, pressure transducers, control valves, and Dowtherm manifolds are mounted behind the rear access plate (Figures 3-10 and 3-11). After cooling, the sample gas, now at 232°C (450°F), is transported through a heat-traced line to the flow control oven.

3.1.1.2 Dowtherm Coolant System

The sample conditioning heater/cooler in the front probe assembly receives working fluid from the Dowtherm system, shown in Figures 3-12 and 3-13. This system maintains the Dowtherm temperature at about 232°C (450°F). The system consists of a pump, heater, heat exchanger, flowmeter, transfer lines, and a primary receiver/deareator tank with a Dowtherm surge/supply tank and an air receiver tank. Dowtherm can be quickly heated or cooled by selected valving arrangements. The pressure can be controlled to raise the boiling point. Heat flux can be controlled with a flowmeter. The self-contained unit also has mechanical instrumentation for flowrate, system pressure, temperature, and fluid level for process monitoring.

3.1.1.3 Hydraulics

Probe insertion, extraction, and precise positioning is accomplished by hydraulic cylinders. These drive the telescoping probe

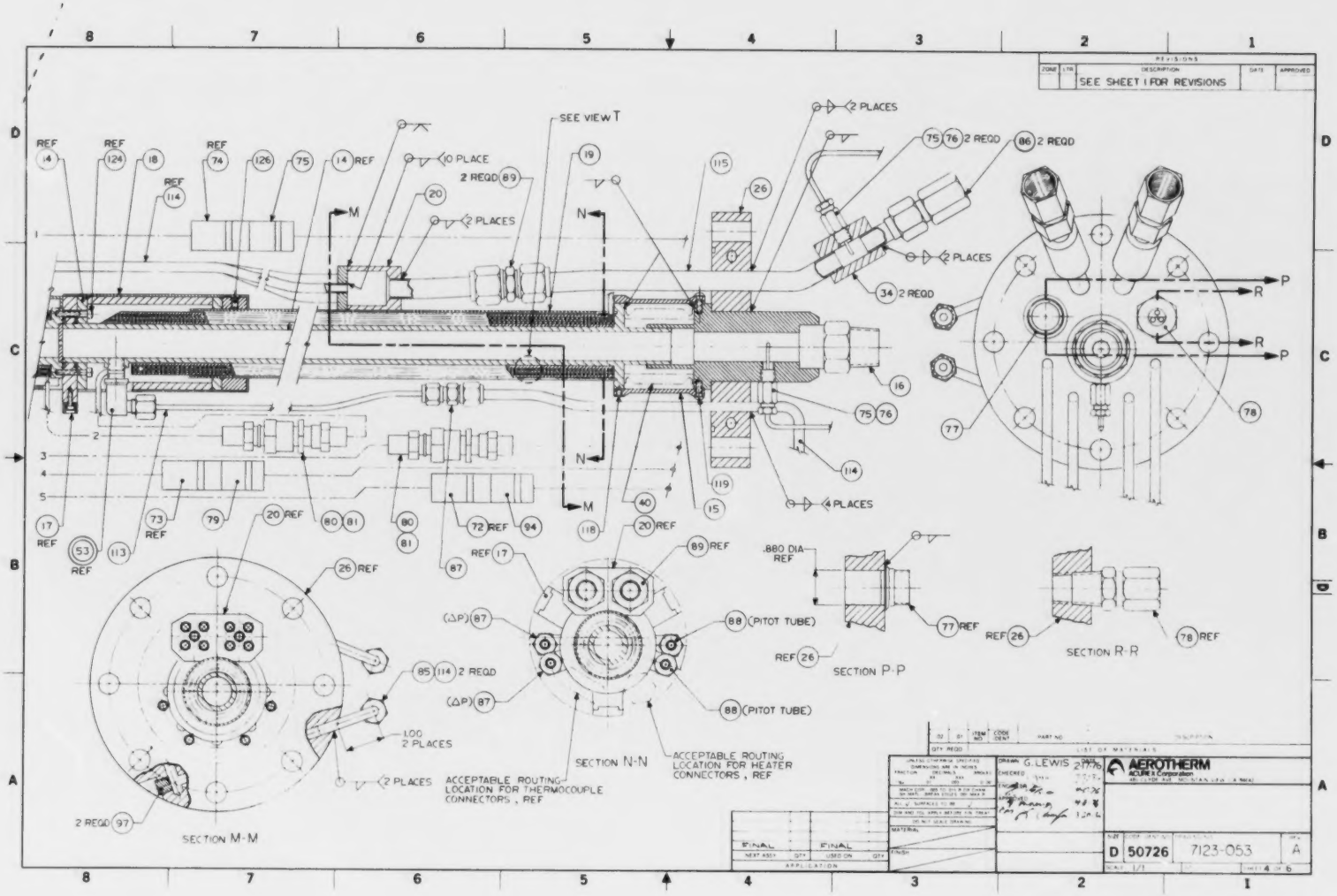


Figure 3-9. HTHP probe/side view/back half.

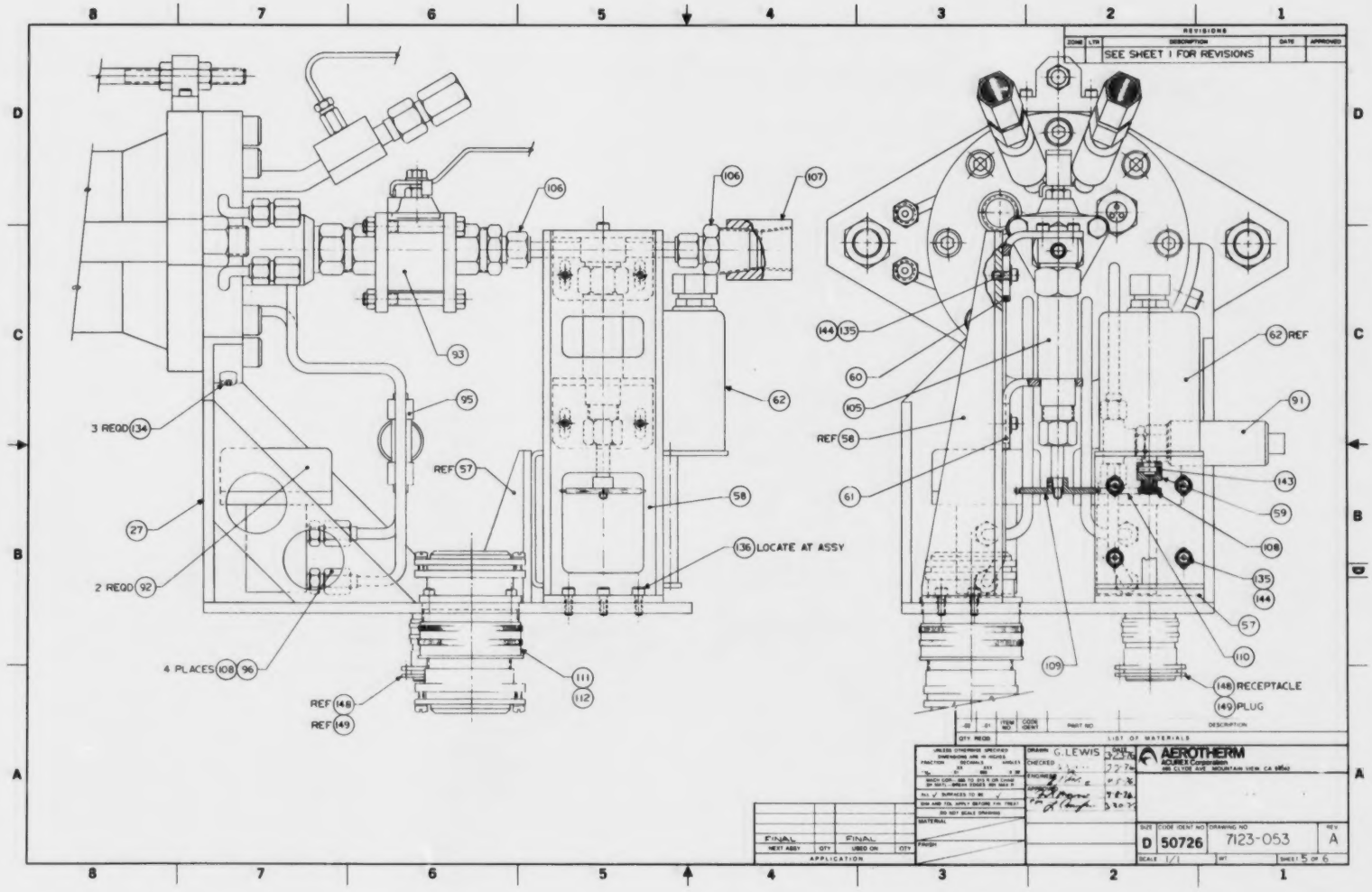


Figure 3-10. HTHP probe rear access plate and controls/side and end view.

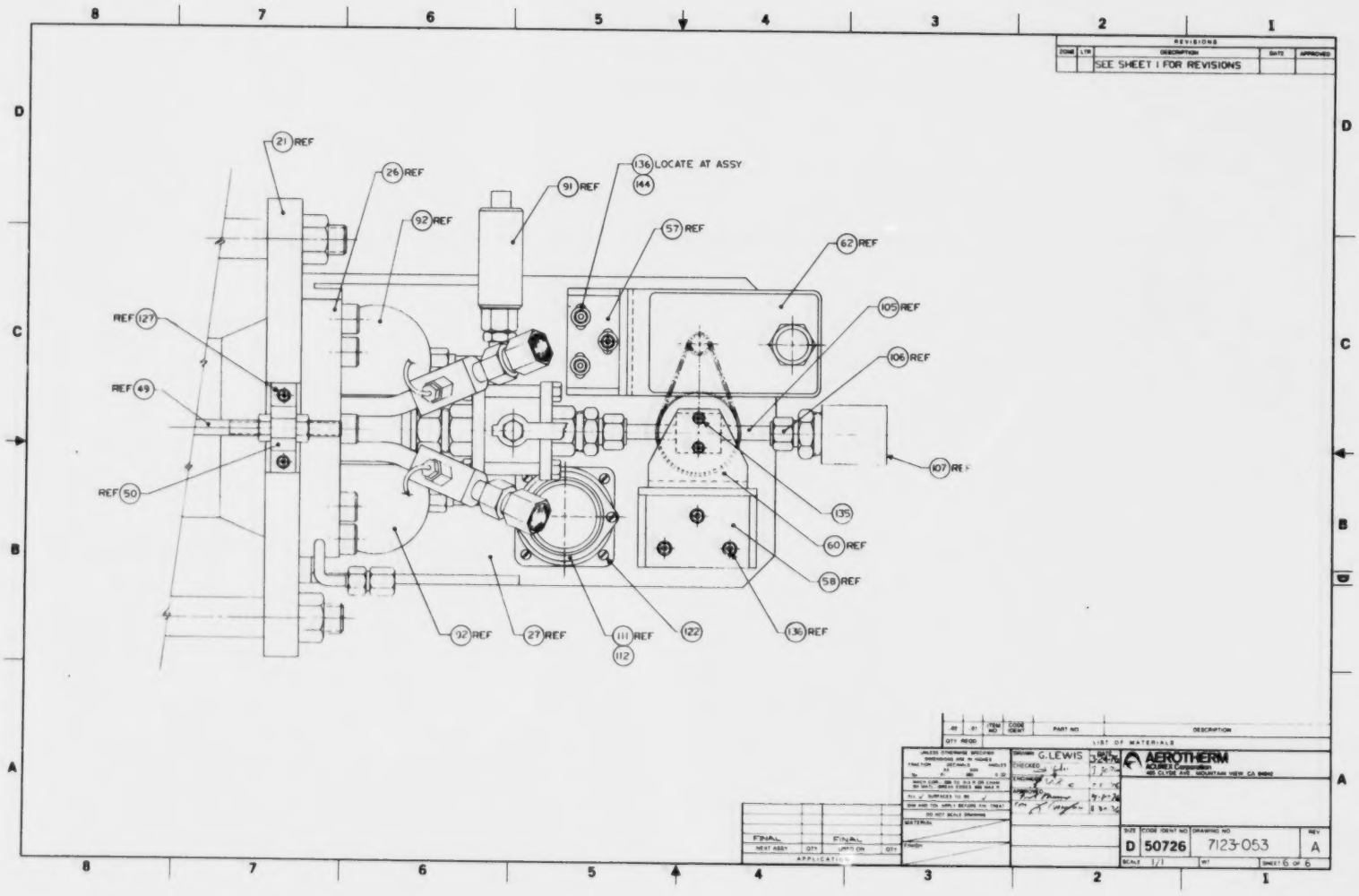


Figure 3-11. HTHP probe rear access plate and controls/top view.

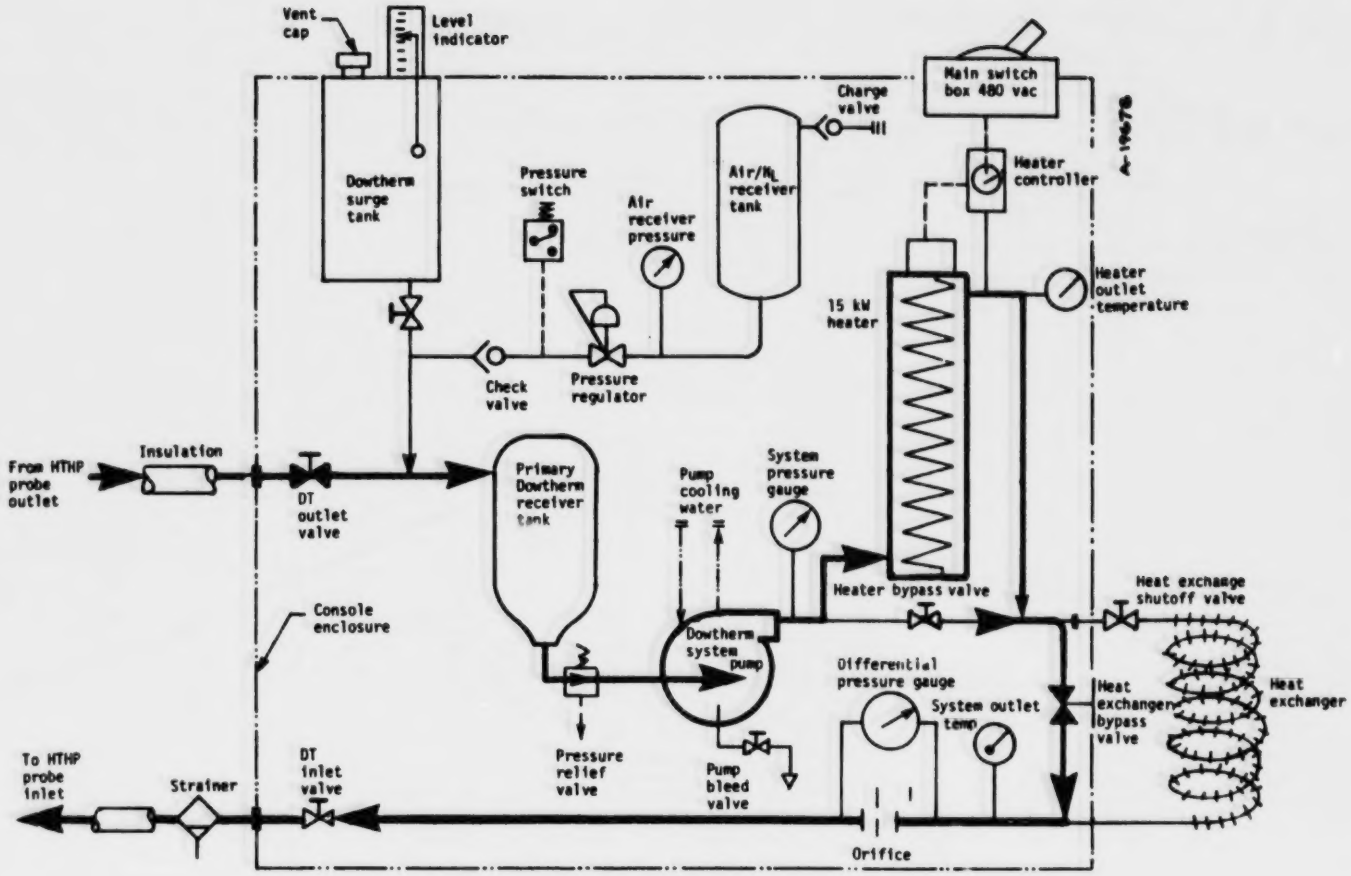


Figure 3-12. Dowtherm system.

Figure 3-13. Dowtherm console.

housing, which, in turn, is driven by a hydraulic pump powered by an electric motor. Solenoid valves plumbed in line with the hydraulic supply lines are used to control the hydraulic cylinders. These operate by remote switches on the probe control console. Fail-safe devices have also been incorporated to avoid inadvertantly traversing the probe. Probe positioning in the process duct can be automatically repeated by setting cams prior to testing.

3.1.1.4 Flow Control Oven

Sample gas at duct pressure exits the throttling valve of the probe and enters the flow control oven. This oven contains a back pressure regulator to reduce sample gas pressure to 172 kPa (25 psig), an adjustable-choked orifice for flow measurement, a heater, and a flow control valve, as shown in Figures 3-14 and 3-15. Excess sample gas is vented to the atmosphere. The balance is routed to the impingers and organic train at a known pressure, flowrate, and temperature. The oven was run at 232°C (450°F) for this series of tests to avoid condensation of selected sample gas constituents. Controls and instrumentation are remotely mounted in the gas train control console which is connected by an umbilical cord containing the various pressure, power, and temperature lines.

3.1.1.5 Organic Module

An organic module was used to collect trace organic gas constituents. It cools the sample gas to 20°C (68°F) and collects organic vapors in a porous polymer granular sorbent bed. Rohm-Haas XAD-2 chromatographic packing material -- chemically cleaned prior to testing -- was the sorbent used. Cooling and conditioning is accomplished by a single and double concentric shell heat exchanger, and by water

Preceding page blank

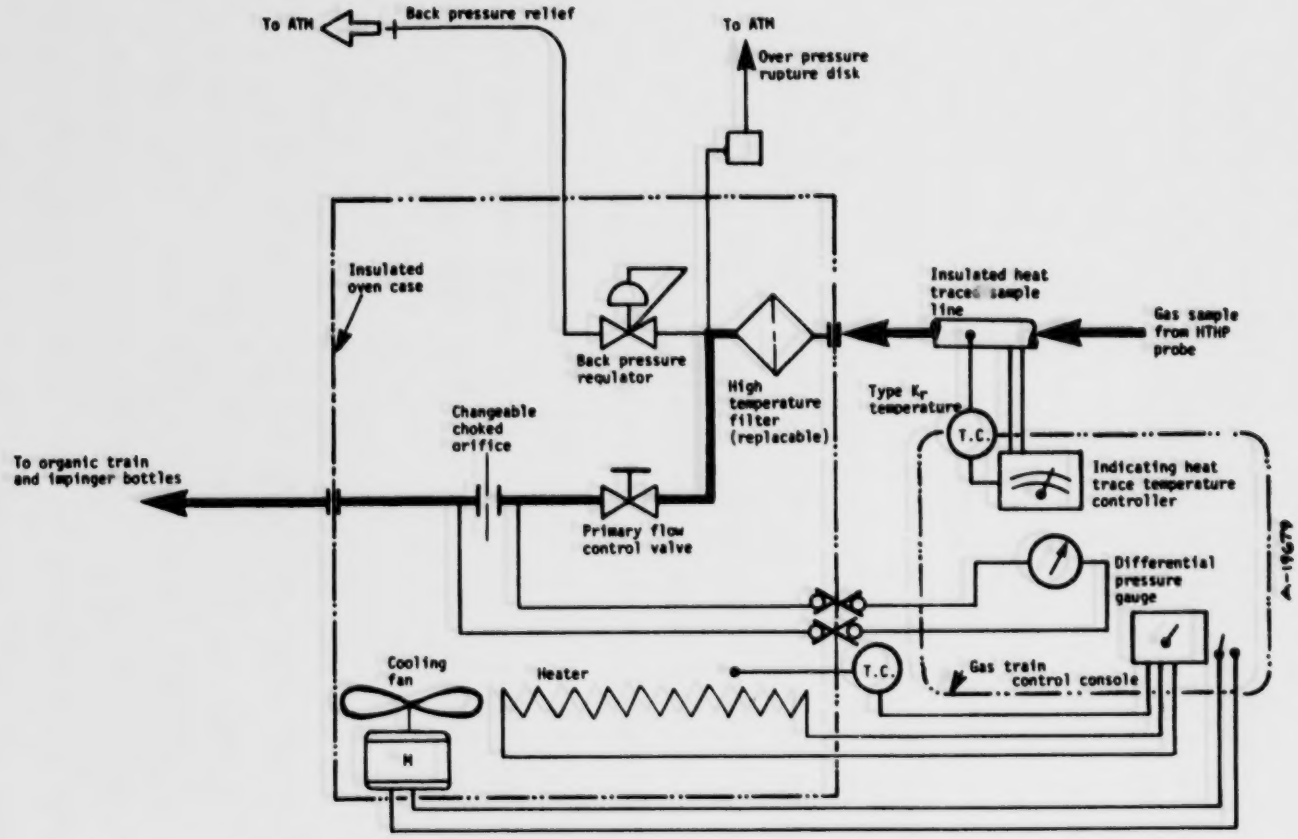


Figure 3-14. Gas sampler train — flow control oven.

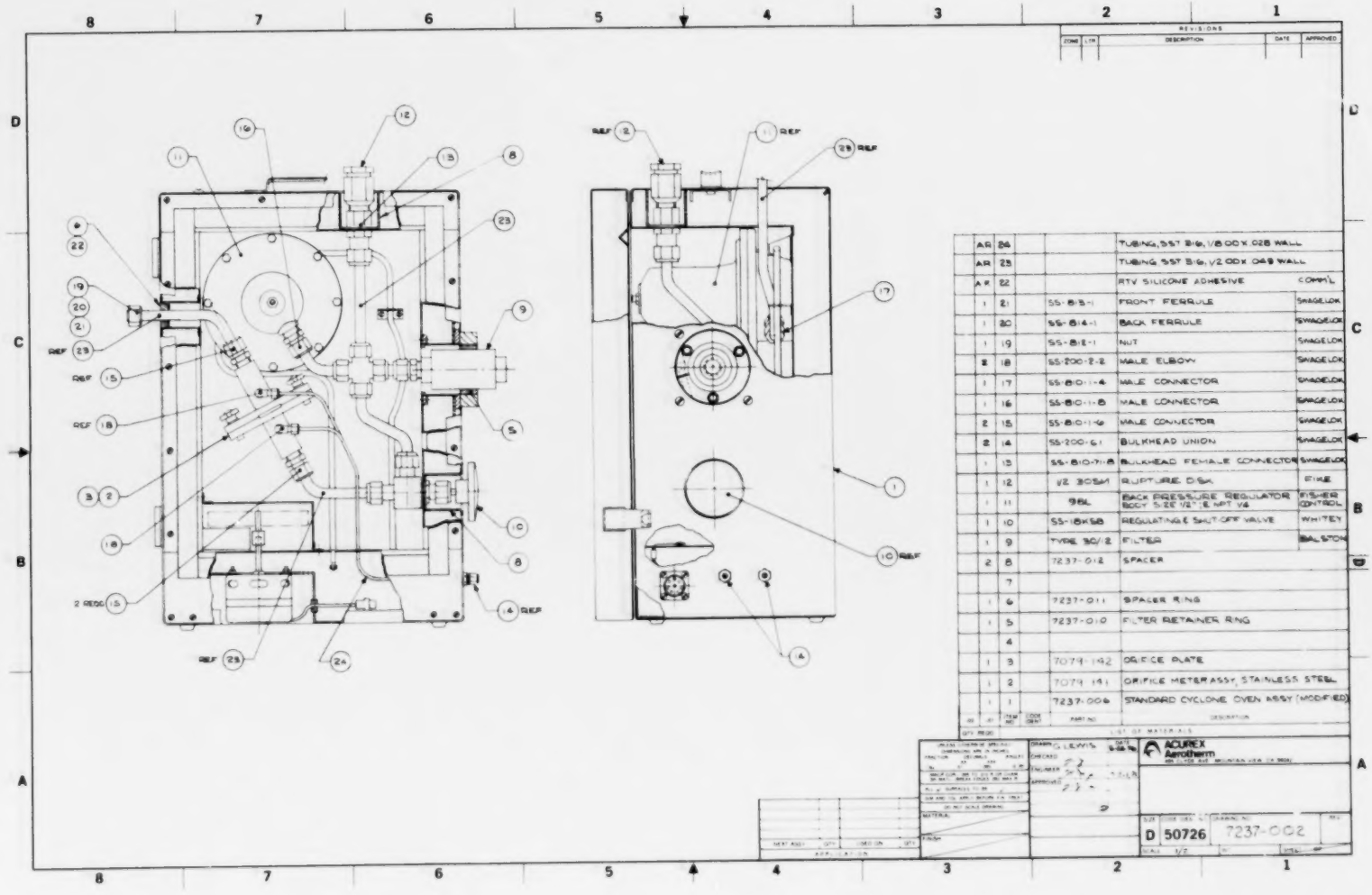

REVISIONS
ZONE LTR DESCRIPTION DATE APPROVED
AR 26 TUBING, SST 316, 1/8 OD X .028 WALL
AR 25 TUBING, SST 316, 1/2 OD X .049 WALL
AR 22 RTV SILICONE ADHESIVE COMM'L
1 21 SS-813-1 FRONT FERRULE SWAGELOK
1 20 SS-814-1 BACK FERRULE SWAGELOK
1 19 SS-812-1 NUT SWAGELOK
2 18 SS-200-2-2 MALE ELBOW SWAGELOK
1 17 SS-810-1-4 MALE CONNECTOR SWAGELOK
1 16 SS-810-1-8 MALE CONNECTOR SWAGELOK
2 15 SS-810-1-16 MALE CONNECTOR SWAGELOK
2 14 SS-200-61 BULKHEAD UNION SWAGELOK
1 13 SS-810-71-8 BULKHEAD FEMALE CONNECTOR SWAGELOK
1 12 1/2 3054M RUPTURE DISK FIKE
1 11 98L BACK PRESSURE REGULATOR BODY SIZE 1/2" E NPT V4 FISHER CONTROL
1 10 SS-18KS8 REGULATING & SHUT-OFF VALVE WHITEY
1 9 TYPE 30/2 FILTER BALSTON
2 8 7237-012 SPACER
7
1 6 7237-011 SPACER RING
1 5 7237-010 FILTER RETAINER RING
4
1 3 7079-142 ORIFICE PLATE
1 2 7079-141 ORIFICE METER ASSY, STAINLESS STEEL
1 1 7237-006 STANDARD CYCLONE OVEN ASSY (MODIFIED)
QTY REQD ITEM NO. PART NO. DESCRIPTION
LIST OF MATERIALS
DRAWN G. LEWIS
CHECKED
ENGINEER
APPROVED
MATERIAL
ACUREX Aerotherm
D 50726 7237-002

Figure 3-15. Oven assembly.

circulating through the impinger ice bath. The water is either heated or cooled automatically to the preset system temperature. Condensation is expected to occur and a valve is available for decanting the sample. This unit is identical to that used in the Acurex commercially available Source Assessment Sampling System. The organic cartridge and the organic module assembly are shown in Figures 3-16, 3-17, and 3-18 respectively.

3.1.1.6 Trace Metal Collectors

A train with three high-volume glass impingers followed by a silica gel dryer was used to collect trace metals. The impingers were charged with the following oxidizing reagents as prescribed by the IERL-RTP Procedures Level 1 Environmental Assessment Manual (Reference 1)

Impinger	Solution
No. 1	6M - H_2O_2
No. 2	0.2 M $(NH_4)_2 S_2O_8$ + 0.02 M $AgNO_3$
No. 3	0.2 M $(NH_4)_2 S_2O_8$ + 0.02 M $AgNO_3$
No. 4	Indicating Type Silica Gel

Figure 3-19 is a photograph of the trace element impingers along with the organic module and flow control oven as they appear assembled for a test run. Figure 3-20 shows a schematic of the organic module and impinger bottle assembly.

3.1.1.7 Controls

The sampling system includes instruments for measuring temperatures and pressures in the process duct, the gas sample, system heaters and coolant. The system has controls for sample flowrate, traverse drive, heaters, coolant pump and purge gas. Sample flowrate was controlled by a motor-actuated valve located at the probe exit. A

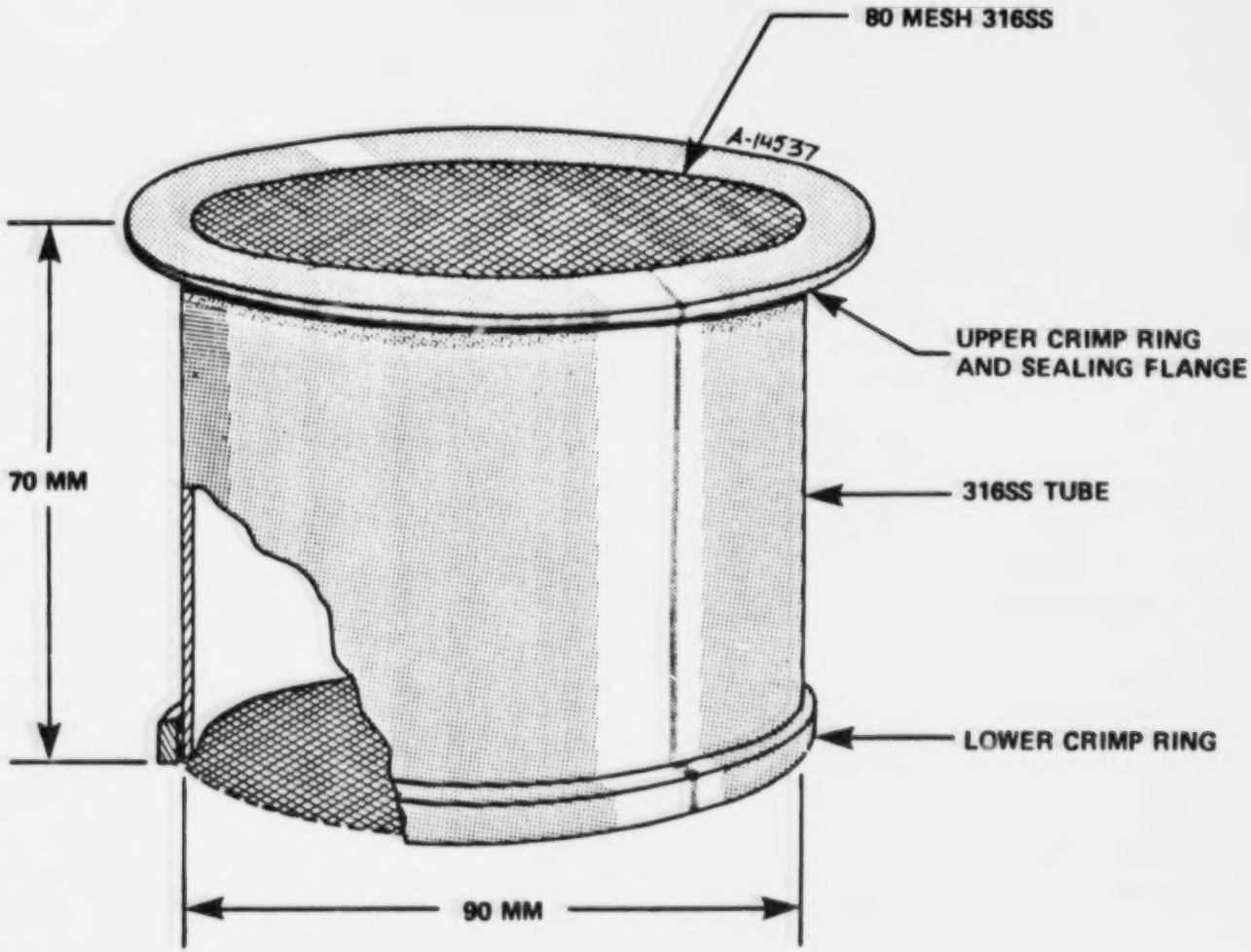


Figure 3-16. Organic cartridge.

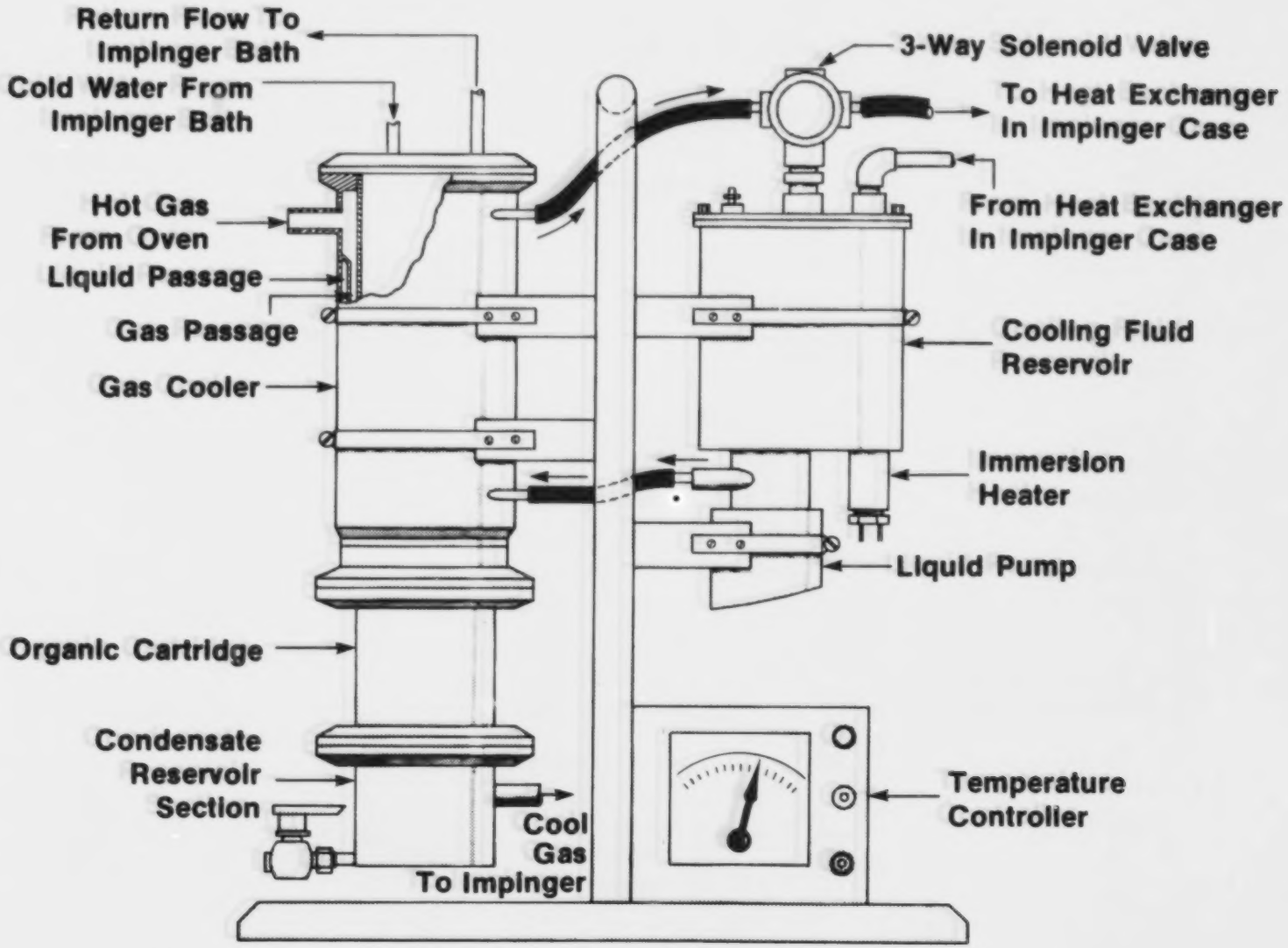


Figure 3-17. Organic module.

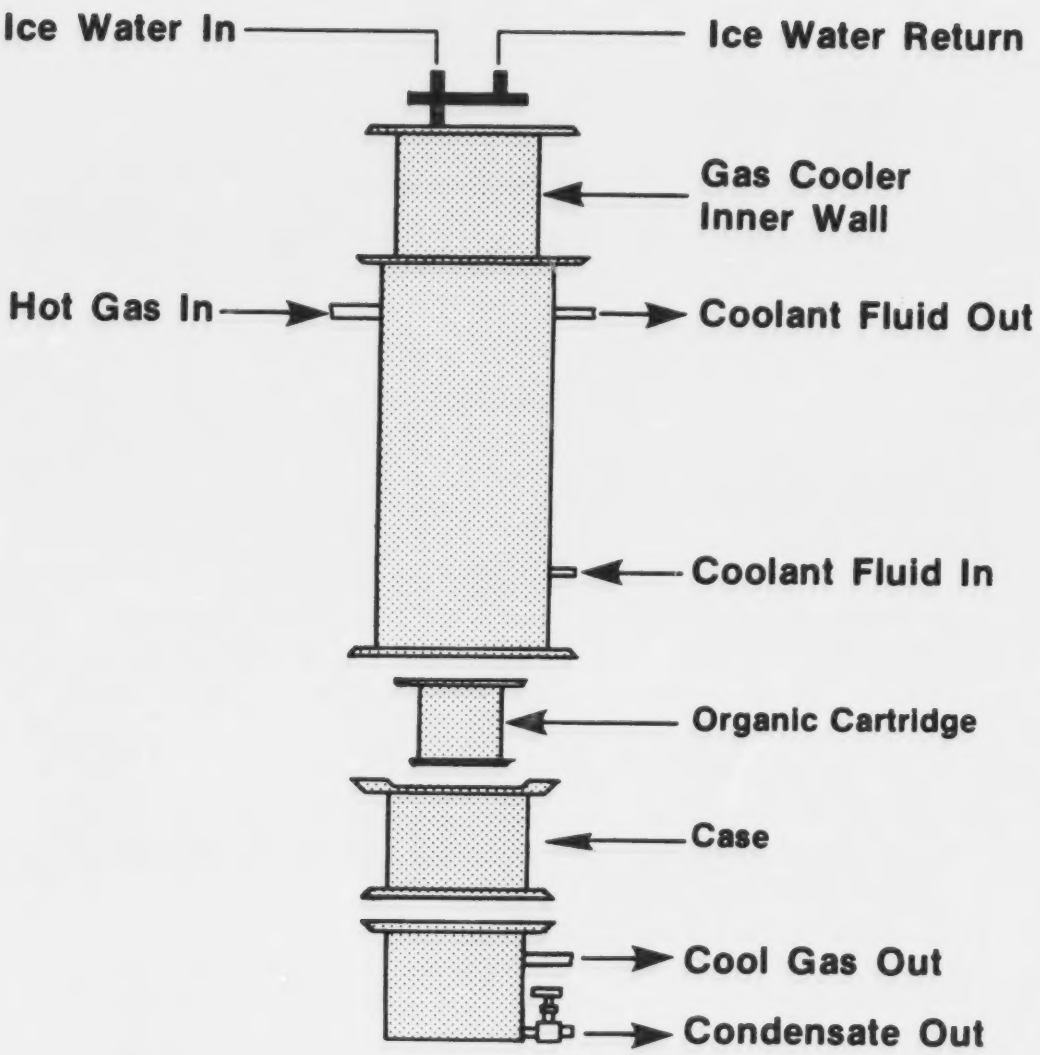


Figure 3-18. Organic module — exploded view.

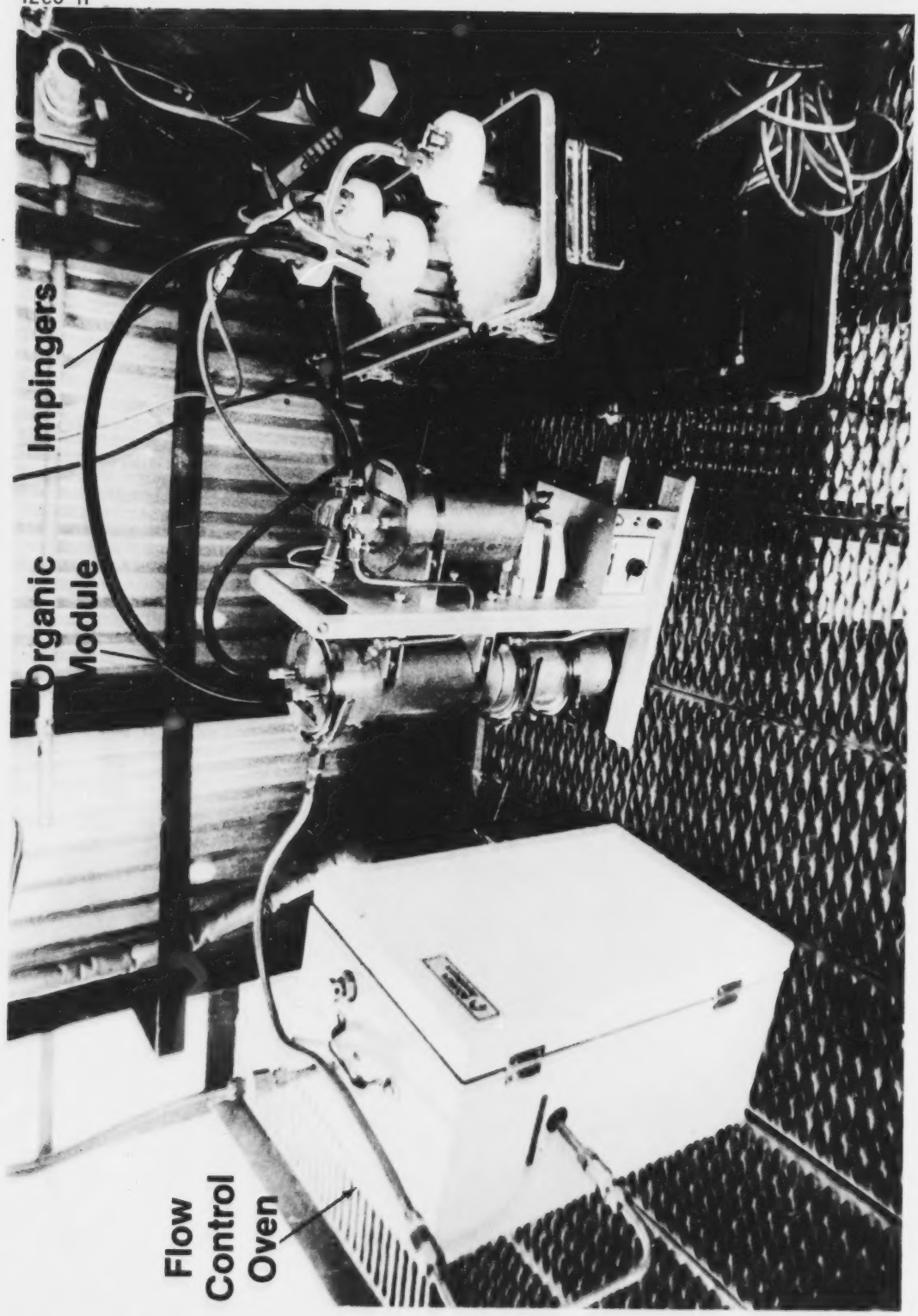


Figure 3-19. Flow control oven and gas train.

Preceding page blank

Preceding page blank

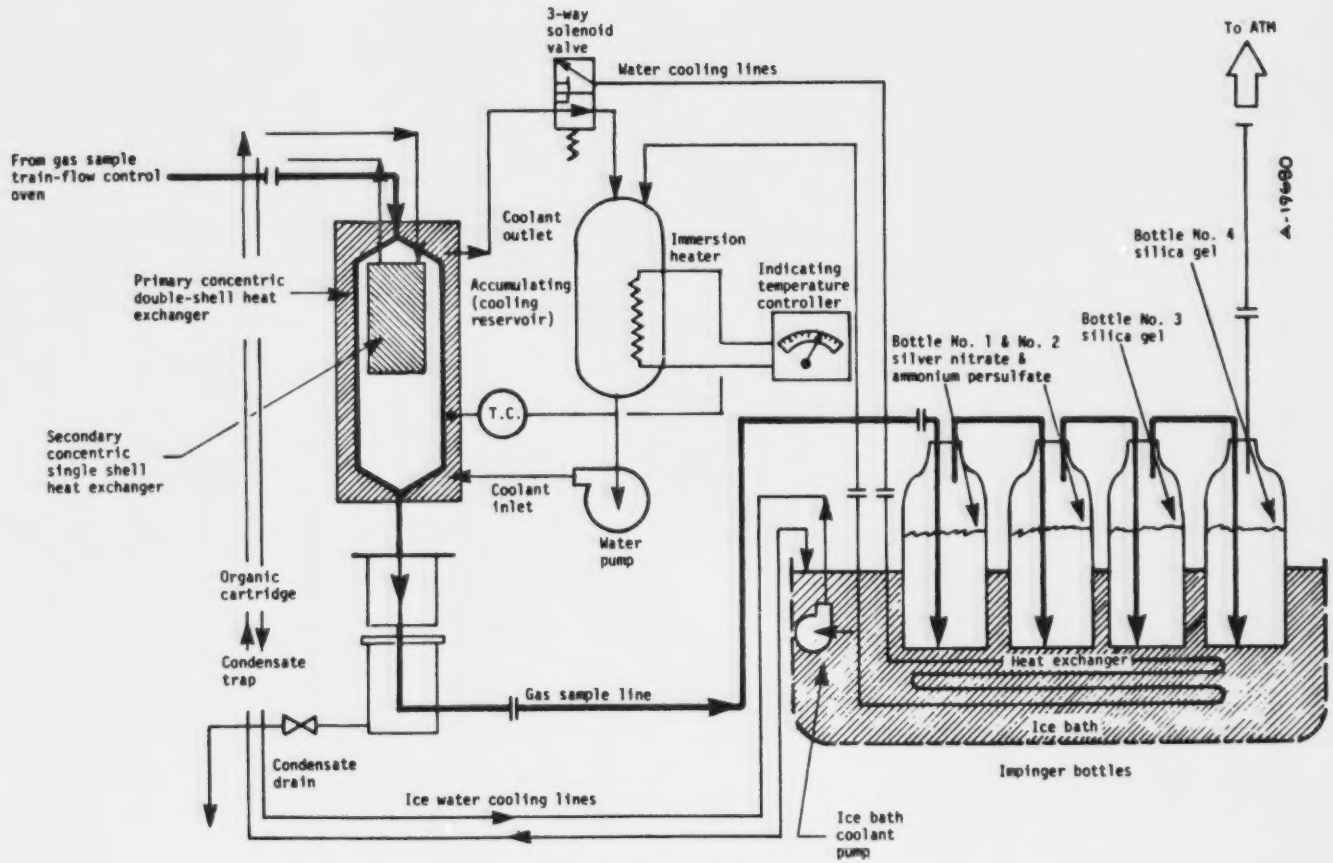


Figure 3-20. Organic module and impinger bottles.

redundant manual valve preceeded the automatic valve for safety purposes. The probe traverse drive was hydraulically actuated from a control console. All heaters were thermoscatically controlled. The coolant pump was manually operated by a switch located on the Dowtherm console. The nitrogen purge gas was supplied by Exxon and controlled by a single stage regulator mounted adjacent to the probe housing. Most of the instrument readouts and controls are housed in the two portable control consoles, shown in Figure 3-21. Control, power and instrument connections are made by multi-pin connectors and umbilical cords.

3.1.2 Phase II -- Condensation Tests

Following the system demonstration tests, a second series of sampling operations was conducted at the Exxon Miniplant. The purpose of these tests was to investigate the effect of sample cooling on measured particulate mass and composition. There was specific concern that trace elements in vapor form might condense to the solid state between process temperature and pressure conditions and the particulate collection temperature and pressure.

Of particular interest to process developers are the more common corrosive alkali metals, sodium and potassium. For these tests, the sampler was configured to collect particulate at process temperature (see Figure 3-22), so trace element condensation occurring within the sampling system could be investigated. First, particulate would be filtered at process conditions, then the sample could be cooled and filtered to collect condensation products. The trace element concentrations could be analyzed and compared for significant differences between the hot and cold filters.

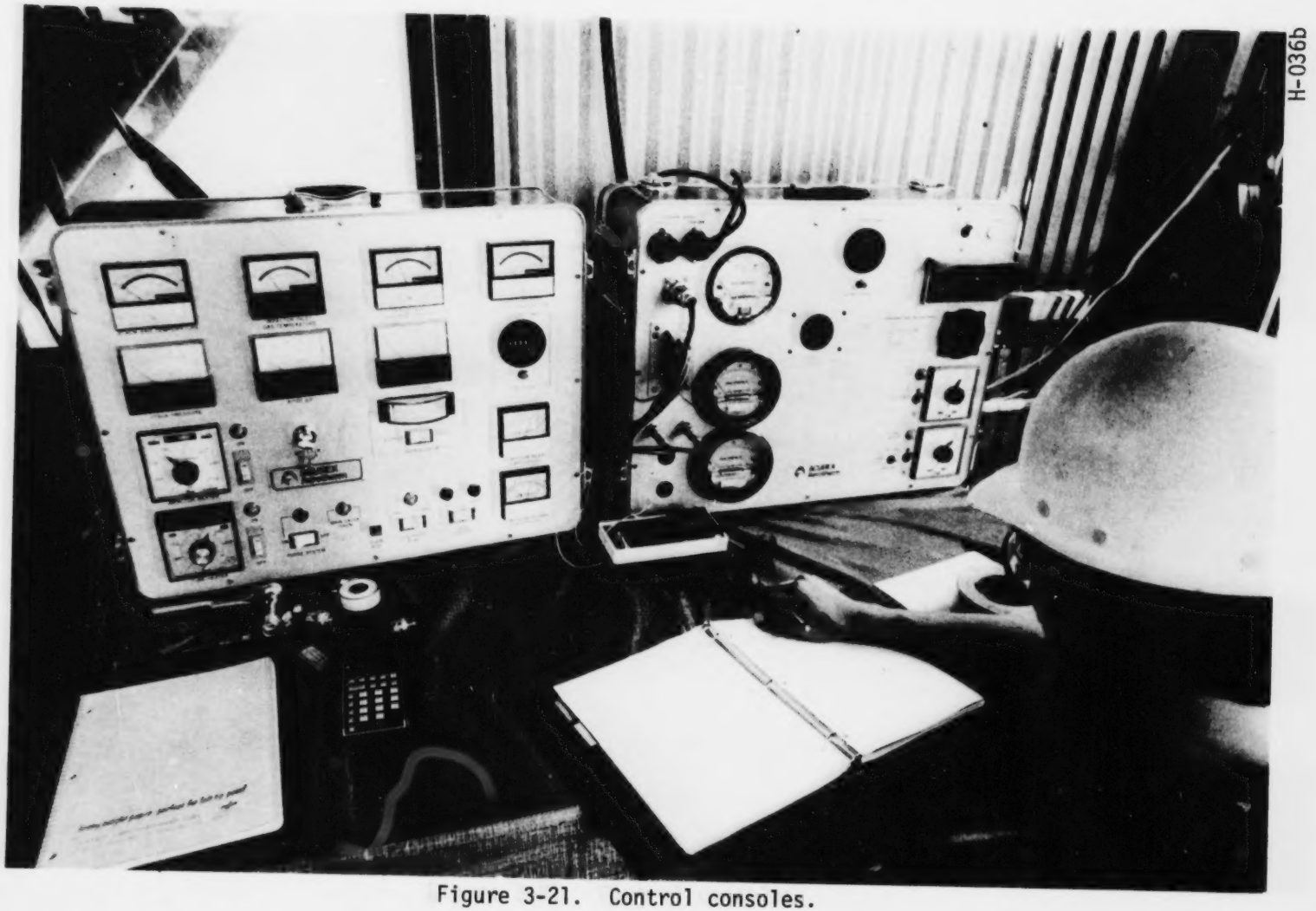


Figure 3-21. Control consoles.

Control consoles.

Preceding page blank

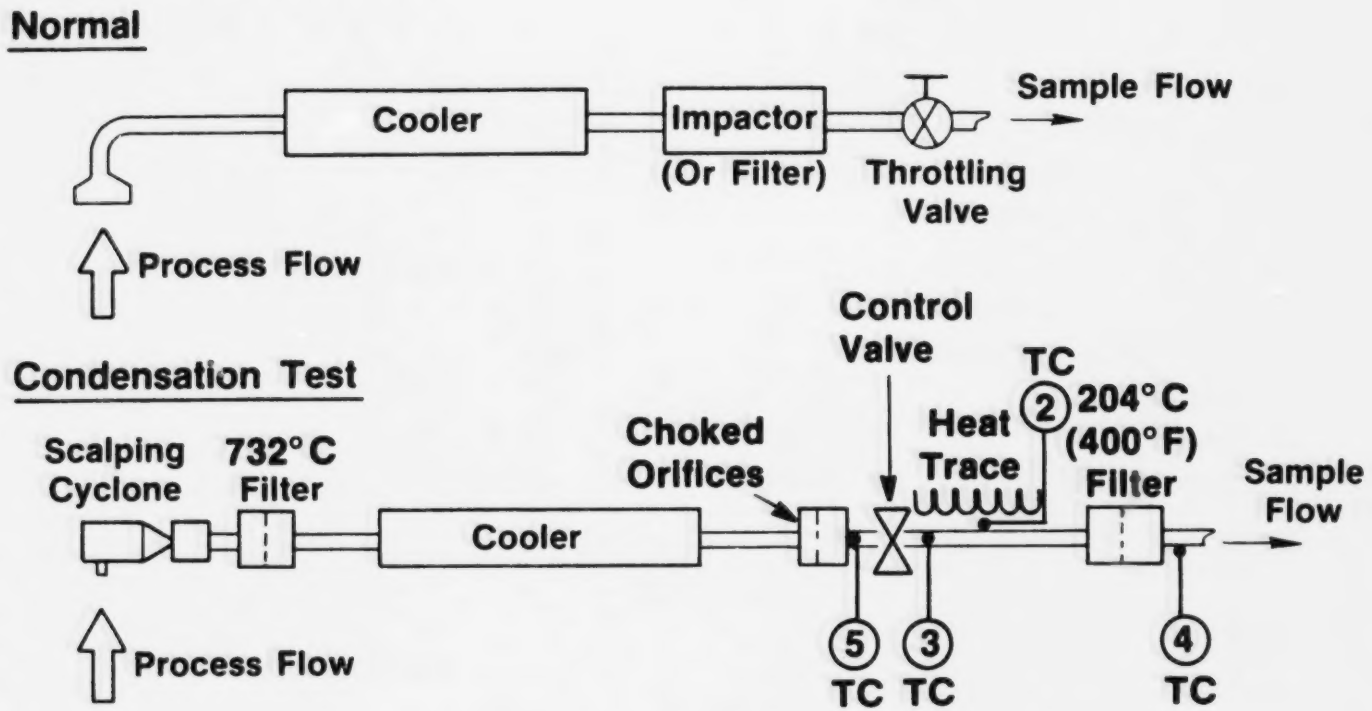


Figure 3-22. Probe configurations.

The individual components of this sampling train, where different from the standard configuration, are discussed in the following sections. It should be noted that since this test series was to study condensation phenomena only, no provisions for sampling trace organics or trace elements by the impinger method were included. After final filtering through the cold filter, the sample gas was simply vented to the atmosphere.

3.1.2.1 Scalping Cyclone

The cyclone (shown in Figure 3-23 and 3-24) at the front of the probe was designed and fabricated by Southern Research Institute to operate at stream ccnditions. It was made of 6AL-4V titanium alloy with a 26-μm thick gold plate for corrosion resistance. Because of the high temperature, it was designed with flat faced flanges and bolted with titanium nuts and bolts.

The cyclone's nominal D_{50} cutpoint is 0.3 μm (aerodynamic diameter) at 4.38×10^{-5} m^3/sec (1.01CFM) at standard temperature and pressure. SRI was unable to predict the D_{50} cutpoint at stream temperature and pressure.

3.1.2.2 Front-End Filter (Saffil Alumina)

The front end filter element is made from Saffil alumina fiber matt. Saffil alumina is a ceramic fiber material that Acurex is currently testing for high-temperature baghouse filters. This material seems to offer excellent temperature resistance and effective filtration, but its performance has not yet been fully characterized. Its performance in the condensation tests (Phase II) was quite good, particularly with a two-filter "sandwich". The filter matt is sandwiched between two 47-mm 316 SST screens stitched together with 316 SST wire.

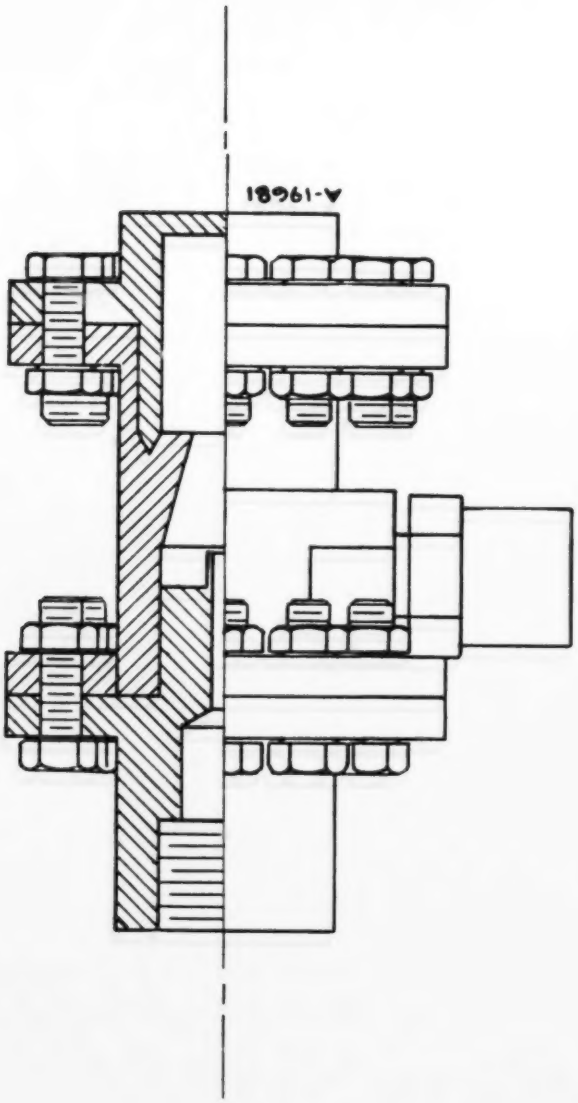


Figure 3-23. Scalping cyclone assembled.

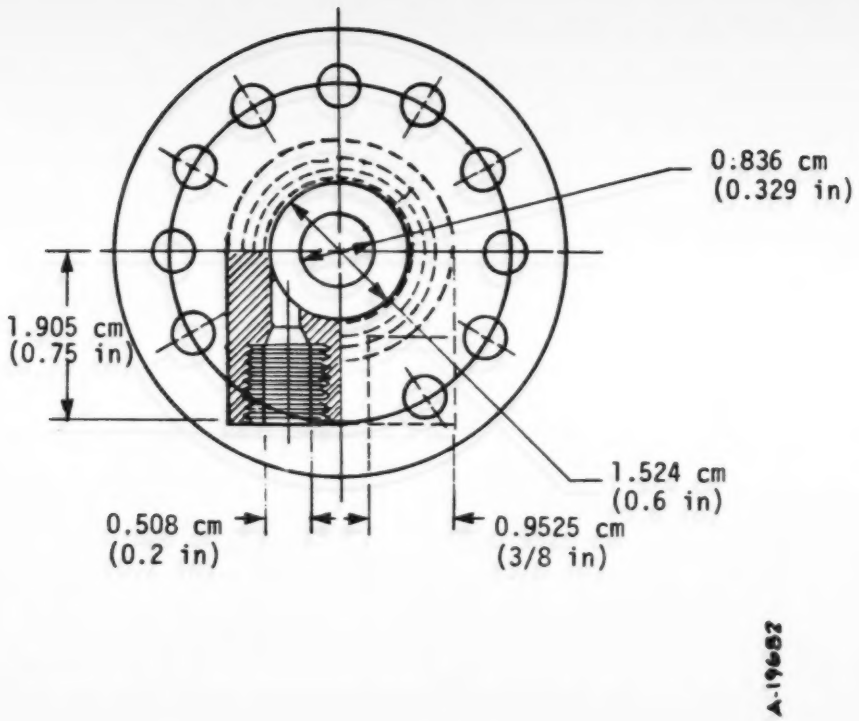


Figure 3-24. Scalping cyclone disassembled.

The filter housing is made from 316 SST and uses flat face bolted flanges for sealing.

3.1.2.3 Sample Conditioning

The primary requirement of the sample conditioning system is to reduce the temperature and pressure to 204°C (400°F) and 101 kPa (1 atm). A further requirement of the system is to not let the temperature drop significantly below 204°C (400°F).

To accomplish this, the sample undergoes the following process:

- Cooling from stream conditions to 232°C (450°F) in the existing Dowtherm cooling section
- Transport through a bypass sleeve in the impactor housing section because it is important to keep the velocity high to keep any condensates from settling
- Reheat to approximately 300°C (570°F). This is necessary because sonic throttling (done in the next section) will cool the gas about 80°C (170°F) at the throat (the temperature will then recover back to 300°C (510°F)). Heating is done by running the gas through an annular passage past electrical heaters, as shown in Figure 3-25. The gas is passed through the annular passage because a high velocity is required to get the necessary heat transfer coefficient.
- Pressure drop from stream pressure to ambient occurs through three critical orifices. This is done to avoid supersonic conditions downstream of each throat and hence very large local temperature drops. The orifices were sized such that the velocity downstream of each throat was slightly supersonic at the design flowrate.

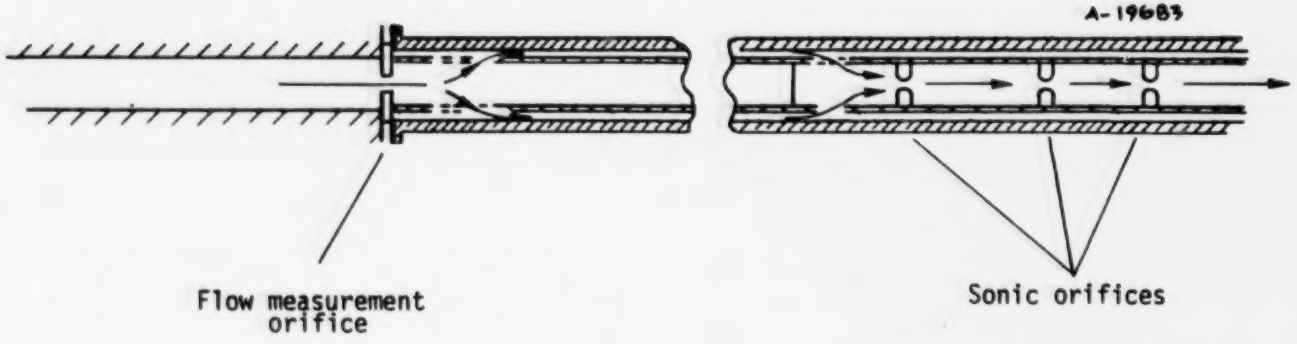


Figure 3-25. Transport tube.

- Cooling from 300°C (570°F) to 204°C (400°C). This is done between the exit of the probe and the rear-end filter housing through natural air convection. The tube is heat traced to give a final temperature control.

3.1.2.4 Final Filter Gelman Micro-Quartz Glass Fiber

The final cleanup filter was a 47-mm Gelman microquartz glass fiber filter. It has a minimum 99.9-percent retention of 0.3 m particles. It was selected because of its low trace metal content.

3.2 EXXON MINIPLANT TEST FACILITY

This section describes the Exxon Miniplant facility itself and the deployment of the sampling system in the facility during both series of sampling operations.

The Miniplant is a pilot-scale pressurized, fluidized bed combustor operated by the Exxon Research and Engineering Company at Linden, New Jersey. The PFBC process is being developed as a more efficient and cleaner method of burning coal. A sketch of a typical PFBC system is shown in Figure 3-26. Coal, along with limestone or dolomite, which act as SO_2 sorbents, is injected into the bottom of the pressurized boiler. Coal is burned in the limestone bed which is fluidized by the incoming combustion air. Sulphur dioxide formed in the combustion process is removed by the limestone bed. Steam coils immersed in the fluidized bed remove some of the heat of combustion and maintain the bed temperature in the range of 816°C (1500°F) to 927°C (1700°F). Steam thus generated operates a steam turbine. The desulphurized flue gas passes through a particulate removal system and is then expanded across a gas turbine. The particulate removal system must reduce the particulate loading down to levels sufficiently low to protect

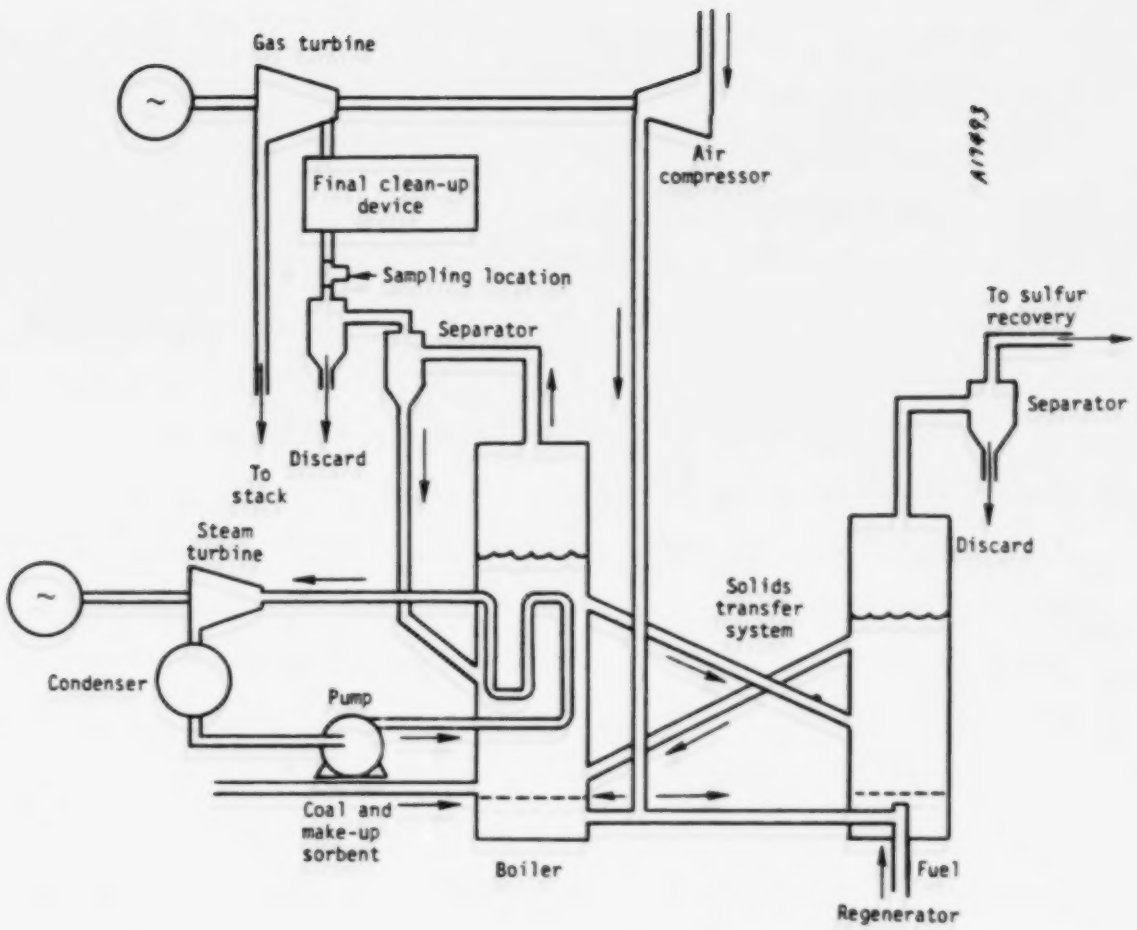


Figure 3-26. Pressurized fluidized bed coal combustor system.

the gas turbine and meet current pollutant emission standards. The Miniplant facility does not presently include a final gas cleanup device or turbines.

The Miniplant facility consists of the combustor tower and control building. The combustor is a four-story structure, with platforms at each level. Stairways connect the platforms. A crane on the top level is available for moving large equipment. The control building includes a laboratory area.

For both test phases, the sampling location was downstream of the secondary cyclone (particulate removal device), as indicated in Figure 3-26. At this location, there is a specially constructed duct section with a sampling port. The sampling port has a 10.2-cm (4-inch) 136-Kg (300-pound) pipe flange which interfaces with the sampling system access valves. The duct diameter at the sampling location is 25.4 cm (10 inches).

The sampling location was physically located at the top of the combustor tower. When installed, the probe assembly was horizontal, about 1.22 m (4 feet) above the platform (see Figure 3-27). The coolant, console and hydraulic pump were also placed on the top platform, near the probe assembly. The control consoles and gas train equipment were set up one floor below, where a partial enclosure gave some weather protection.

The route between the laboratory area and the sampling location included four flights of stairs and about a 30.5 m (100-feet) walk. The sample probe assembly was hand-carried along this route before and after each sampling run. Probe cleaning, assembly, disassembly and sample removal were all done in the laboratory. The lab facility had an analytical balance, oven, desiccator and other equipment used in sampler

Preceding page blank

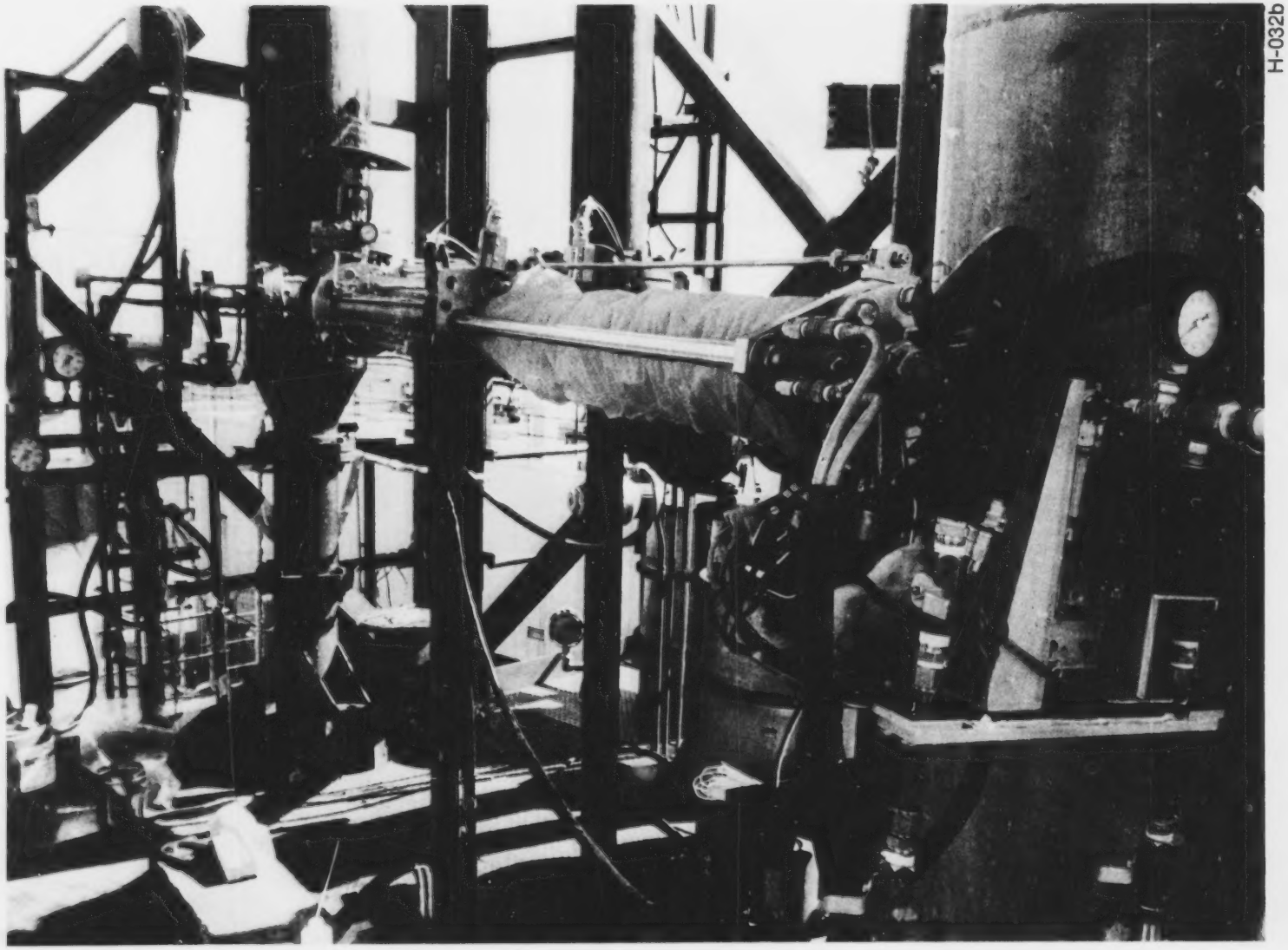


Figure 3-27
HTHP probe assembly
installed at Exxon miniplant.

preparation and sample processing. Labware and materials were supplied by Acurex.

The Exxon facility provided a number of utilities supporting the sampler operation. Power connections, water, and nitrogen supplies are summarized in Table 3-2. In addition, Exxon supplied technician support during equipment setup and disassembly.

TABLE 3-2. UTILITY REQUIREMENTS

Electrical:	
480 VAC, 3 phase, 40 amp	1 line
115 VAC, 15 amp	6 lines
Water:	
18.9 L/min (5 gpm), 34.47 kPa (50 psi)	1 line
Pure Nitrogen:	
(flow and pressure required depending on stream conditions)	1 line
• For this test, about 8.76×10^{-4} Nm^3/sec. (2 scfm) at 861.8 kPa (125 psi)	

Preceding page blank

SECTION 4

TEST DESCRIPTION

This section describes assembly and operating procedures for the sampling equipment and some of the significant events which occurred during the two test phases. The narrative of events in each phase is divided into four sections: procedures, pretest activities, sampling runs, and post-test activities.

4.1 PHASE I -- DEMONSTRATION TESTS

4.1.1 Procedures

Equipment setup and operation was done according to a formal procedure which defined proper installation of access valves and probe housing, probe setup and assembly, system preparations for testing, test sequence, shutdown and sample removal.

In several cases, decisions were made in the field to change predefined procedures. For example, the exposure and limited space on the sampling platform made impactor removal at the sampling location impractical. The entire probe was carried to the lab for disassembly.

In precleaning the sampling equipment, the procedures in the IERL-RTP Procedures Manual for Level 1 Environmental Assessment were followed with one exception; the nitric acid passivation of some internal surfaces of the probe, organic module and flow control oven was omitted

because large acid containers were not available. Sample removal and post-test cleaning also followed Level 1 procedures.

4.1.2 Pretest Activities

Pretest activities included planned unpacking, setup and checkout, plus fixing several problems with the facility and sampling system. The test preparations were completed between March 22 and March 30. Heavy equipment was installed with the help of Exxon personnel. For the nitrogen purge gas, Exxon provided a connection to the facility nitrogen supply. Exxon also assisted in making a support for the cantilevered probe housing.

4.1.3 First Run

For the first test run, the sampling system was set up using the thimble filter particulate collector and gas train equipment for the purposes of obtaining grain loadings. Following preheating, the duct access valves were opened and the sampling probe inserted into the duct stream. The sample flow control valve was opened until the flow orifice indicated a sample flow of 0.75 acfm at nominal particulate collector conditions. When flow conditions were established, the gas train flow control valve was opened, diverting total sample flow through the organic module and impinger train. Sampling continued for 30 minutes. Instrument readings during the test run are listed in Table 5-2. At the end of the test run, the motor driven sample flow valve was left open and the sample flow was shut off using the manual ball valve. The probe was then withdrawn and gate valves were closed. After cooldown, the probe assembly was removed. The probe and gas train were taken to the lab area for sample recovery and cleaning.

4.1.4 Second Run

For the second test run, the cascade impactor was used for particulate collection. Since this was to be a very short test with a small amount of gas sample collected, the gas train equipment was not used. Based on estimated particle concentration and impactor capacity, the maximum sampling duration was estimated to be between 30 seconds and 1 minute. For this test run, 30 seconds was chosen. To achieve proper sample flow as soon as possible, the motor driven control valve was left at the same setting as the earlier filter run, and on-off control accomplished with the manual ball valve. As soon as the probe reached the in-stream position, the sample flow was started. No attempt was made to adjust flow while sampling. After 30 seconds, sample flow was stopped with the ball valve, the probe was withdrawn and access valves closed. After cooldown and probe removal, the probe assembly was carried as carefully as possible down the combustor tower stairs to the lab. There, the impactor assembly was removed, disassembled and inspected. The amount and patterns of the catch seemed to indicate normal operation of the device (see Figures 5-3). However, on one stage (Stage 4) the substrate shifted slightly, and on another (Stage 7) some of the jets were plugged. After sample removal and cleaning, the probe was ready to be set up for the third and final test run.

4.1.5 Third Run

The third test run also used the impactor for particulate collection and omitted the gas train equipment. Based on the lightly loaded appearance of the 30-second impactor catch from Run No. 2, the duration of this run was increased to 1 minute. The flow control method for this run was identical to Run No. 2. The manual valve was again used

to start and stop sample flow with no attempt to adjust flowrate during sampling. Again the probe was taken to the lab for disassembly and sample removal. The impactor substrates were noticeably more heavily loaded than for the 30-second impactor run (see Figures 5-3 and 5-4). Figures 5-3 and 5-4 also show that the substrates from Stage 7 were partially plugged for these runs. We were unable to clear the jets without risking alteration of the jet diameter. With the completion of sample removal and cleaning of the sampling equipment, the testing phase was finished.

4.1.6 Post-Test Activities

Following test completion, sampling system hardware was packed and stored onsite at Exxon in preparation for the Phase II sampling program. Test samples were brought back to Acurex, where analysis was performed.

4.2 PHASE II -- CONDENSATION TESTS

4.2.1 Procedures

Equipment setup and operation was executed according to an amended formal procedure which was developed from the previous demonstration tests. This defined a proper sequence of assembly of the gate valves, probe housing, ancillary equipment and prescribed probe setup, systems preparations for testing, test sequence, shutdown and sample removal.

As in the previous demonstration tests, all procedures followed the IERL-RTP Procedures Manual for Level 1 Environmental Assessment.

4.2.2 Pretest Activities

Pretest activities consisted of unpacking the probe from Exxon, storage, setup and checkout, and check fitting the modified probe parts. Test preparations were completed between May 21 and May 22. Exxon personnel assisted in the installation of heavy equipment and utilities

supply. The previous nitrogen purge gas, probe housing support, and cooling water lines were used.

The front cyclone and filter housing, choked orifice train, extension tube and rear filter housing were fitted to the outer probe housing for check fitting and traversed through the operating modes. During the first traverse through the access valves, the front cyclone and filter failed to clear the sampling valve seats resulting in a damage to the cyclone nozzle and filter housing. A redesigned filter housing which featured a reduced profile and an integrated filter screen was machined on May 23 with the assistance of Exxon personnel. The assembly was retested May 25 with success.

4.2.3 Runs 1 and 2

For the first test run, the choked orifice train was not used due to an installation problem involving O-ring seal abrasion. Instead, the primary flow control valve was set to the desired flow of 0.0144 m^3/min using the previous demonstration test data results. Following the prescribed preheating, the duct access valves were opened and the sampling probe inserted into the duct stream. The manual ball valve was opened and the flow measured by the existing orifice within the inner probe. Sampling continued for 30 minutes with data taken at 5-minute intervals. The manual ball valve was closed with the flow control valve left in its initial position. The probe was then withdrawn and gate valves closed. The system was then cooled and the probe assembly removed and taken to the lab area for sample recovery and cleaning. During sample recovery, it was noted that the front filter had evidence of particulate blowby. Cleaning solvent rinses were bottled and labeled for analysis.

For the second test run, the choked orifice train was installed by modifying mechanical links for more clearance within the existing transport tube. To prevent mechanical abrasion of the O-ring seals, a rinse solvent mixture was used as a final rinse and lubricant. Both test runs were conducted May 23.

4.2.4 Runs 3 and 4

For Test Runs 3 and 4, the choked orifice train, extension tube, front and rear filters, and the cyclone were used following the identical procedures and test parameters as Run 2. Two saffil alumina filters were "sandwiched" to comprise the front filter in order to minimize particulate blowby. Sample recovery showed little or no sign of particulate blowby on these runs. However, chemical analysis of the cyclone and front filter catch showed significant gold contamination. This was caused by failure of the cyclone's protective gold plating upon extended exposure to the PFBC flue gases. Titanium contamination due to heavy oxidation of the cyclone body was not evident in any of the samples. With the completion of sample recovery and cleaning, the testing phase was finished.

4.2.5 Post-Test Activities

The sampling system hardware was packed for surface shipment back to Acurex. Solvent wash test samples were sent by special surface carrier and test filters were hand-carried back to Mountain View. Packing and packaging were completed May 24.

SECTION 5

DATA AND RESULTS

This section presents the detailed information collected and the results obtained during the complete HTHP sampling system test program at Exxon's Miniplant. This section has been divided into subsections which discuss the individual data and results from each of the two test series, the Phase I -- Demonstration Tests and the Phase II -- Condensation Effects Tests. The Phase II discussion also includes a discussion on the comparative results of both phases of testing.

5.1 PHASE I -- DEMONSTRATION TEST DATA

5.1.1 Test Conditions

Plant operating conditions are listed in Table 5-1. The nominal conditions were identical for all three sampling runs. The facility ran steadily without interruption during the tests.

5.1.2 Instrument Readings

The sampling system includes a number of instruments which measure duct, sample and equipment operating conditions. Readings from these instruments are presented in Tables 5-2, 5-3 and 5-4.

Table 5-2 gives readings from probe assembly instruments. Duct temperature and pressure were typically about 740^{o}C (1360^{o}F) and 860 kPa (110 psig). The drop in temperature from 900^{o}C (1650^{o}F) in the combustor to 740^{o}C (1360^{o}F) at the sampling location is due to normal

TABLE 5-1. TEST CONDITIONS

Run:	#1	#2	#3
Date	3-31-77	4-1-77	4-1-77
Time	3:30 p.m.	10:30 a.m.	2:40 p.m.
Ambient temperature	18°C (64°F)	21°C (70°F)	19°C (67°F)
Bed Conditions			
Temperature	900°C (1650°F)	900°C (1650°F)	900°C (1650°F)
Pressure (gage)	912 kPa (9 atm)	912 kPa (9 atm)	912 kPa (9 atm)
Ca/Sulphur Ratio	1.25	1.25	1.25
Excess Air	30%	30%	30%
Coal	Champion	Champion	Champion
Dolomite	Pfzizer	Pfzizer	Pfzizer
Flowrate - sm^3/min (scfm)	14(544)	14(546)	14(546)
Average Duct Velocity - m/s(ft/sec)	2.0(6.7)	1.9(6.3)	2.0(6.7)

TABLE 5-2. PHASE I PROBE INSTRUMENTATION READINGS

	Time	Elapsed Time (minutes)	Stack Pressure (psig)	Stack Gas Temp °C(°F)	Dowtherm Inlet Temp °C(°F)	Dowtherm Exit Temp °C(°F)	Sample Temp Impactor Inlet °C(°F)	Sample Temp Orifice Inlet °C(°F)	Sample Temp Transport Tube Exit °C(°F)	Sample Flow-rate $m^3/sec \times 10^{-4}$ (acfm)	Impactor Heater Temp °C(°F)	Transport Tube Heater Temp °C(°F)
Run No. 1												
	3:10 p.m.	0	861.8(125)[a]	77(170)	222(431)	216(420)	-	161(322)	-	0(0)	-	232(450)
Insertion	3:22 p.m.	0	861.8(125)[a]	83(190)	218(425)	208(407)	-	167(332)	-	0(0)	-	233(452)
Sample Flow	3:32 p.m.	0	758.4(110)	738(1360)	225(437)	224(435)	-	167(332)	-	0(0)	-	233(452)
	3:35 p.m.	0.1	751.5(109)	732(1350)	225(437)	224(435)	-	178(352)	-	3.45(.73)	-	233(452)
		5.0	758.4(110)	732(1350)	225(437)	224(435)	-	189(373)	-	3.02(.64)	-	233(452)
		10.0	758.4(110)	727(1340)	225(437)	224(435)	-	202(395)	-	3.87(.82)	-	233(452)
		15.0	751.5(109)	716(1320)	225(437)	224(435)	-	210(410)	-	3.92(183)	-	229(445)
		20.0	758.4(110)	716(1320)	218(425)	208(407)	-	216(421)	-	4.01(.85)	-	238(460)
		25.0	758.4(110)	716(1320)	225(437)	224(435)	-	227(441)	-	4.11(.87)	-	238(460)
		30.0	758.4(110)	716(1320)	225(437)	224(435)	-	237(459)	-	4.15(.88)	-	238(460)
Shut-off												
Run No. 2												
Before Insert	10:30 a.m.	0	834.3(121)[a]	91(195)	222(431)	216(420)	-	167(332)	-	0(0)	-	229(445)
Sample Flow		0.5	827.4(120)	738(1360)	222(431)	216(420)	-	107(225)	-	4.25(.90)	-	229(445)
Run No. 3												
	2:58 p.m.	0	861.8(125)[a]	88(190)	222(431)	216(420)	-	164(327)	-	0(0)	-	229(445)
Insertion		0	765.3(111)	738(1360)			-	164(327)	-	0(0)	-	
Sample Flow		0		738(1360)			-		-	0(0)	-	
		0.33		738(1360)			-		-	4.29(.91)	-	
		0.67		738(1360)			-		-	4.20(.89)	-	
		0.83		738(1360)			-		-	4.15(.88)		
		1.0		738(1360)			-		-		-	

TABLE 5-3. GAS TRAIN INSTRUMENT READINGS

	Time	Elapsed Time	Flow-rate $m^3/sec \times 10^{-4}$ (acfm)	Transport Line Temp °C(°F)	Flow Control Oven Temp °C(°F)	Organic Module Temp °C(°F)	Impinger Train Temp °C(°F)	Ambient Temp °C(°F)
Run No. 1			4.72(1.0)[a]					
	3:10 p.m.	0	0(0)	229(444)	232(449)	14(58)	21(70)	18(64)
	3:22 p.m.	0	0(0)	234(453)	-	-	-	
	3:32 p.m.	0	0(0)	-	-	-	-	
Start Flow		0	3.45(.73)	-	-	-	-	
		5.0	3.02(.64)	232(449)	202(396)	21(70)	21(69)	18(64)
		10.0	3.87(.82)	232(449)	218(425)	22(72)	21(69)	18(65)
		15.0	3.92(.83)	231(447)	237(458)	23(74)	21(70)	19(67)
		20.0	4.01(.85)	231(447)	234(454)	22(71)	22(71)	19(67)
		25.0	4.11(.87)	236(456)	230(446)	24(76)	23(73)	18(65)
Stop Flow		27.65	4.15(.88)	232(450)	229(444)	24(75)	23(73)	19(66)

[a]At orifice conditions - see Table 4-2.

TABLE 5-3. GAS TRAIN INSTRUMENT READINGS

TABLE 5-4. ANISOKINETIC CORRECTION FACTORS - PHASE I

Run	Sample Flowrate m^3/sec (acfm)	Nozzle Velocity m/sec (ft/sec)	Estimated Duct Velocity m/sec (ft/sec)	Velocity Ratio	Particle Concentration[a] Correction Factor $\frac{C_{measured}}{C_{true}}$
1	4.0×10^{-4} (0.85)	2.8(9.3)	2.0(6.7)	0.72	0.99
2	4.0×10^{-4} (0.90)	4.0(13.0)	1.9(6.3)	0.48	0.98
3	4.0×10^{-4} (0.90)	3.4(11.3)	2.0(6.7)	0.59	0.99

[a]Calculation per method in Handbook of Aerosols, TID-26608, 1976, Section 5.1-1 and Figure 5-2.

system heat losses. Sample conditioning data showed that the sample gas was cooled below the desired 230°C (450°F) in passing through the unheated particle collection device. In Run 1, with the filter collector, minimum temperature after the impactor was 107°C (225°F). Temperatures did, however, remain above the dewpoint for 97°C (207°F), 930 kPa (120 psig) 6 percent water (although probably not above the H_2SO_4 dewpoint). Consequently, no water condensation occurred during these tests. Correcting the impactor heater malfunction will eliminate the low collection temperatures in future sampling.

The sample flowrate, 380 cm^3/s to 425 cm^3/s (0.8 to 0.9 acfm) at orifice inlet conditions, was maintained within the impactor operating range throughout the test series. This flowrate gave nozzle velocities which were somewhat above duct velocity (anisokinetic). The flowrate was chosen based on the expected stream velocity. Unfortunately, a damaged pitot tube prevented measuring the velocity to confirm the proper flowrate needed to maintain isokinetic conditions. However, for the high gas temperature and pressure, fine particles and low velocities involved, the variance from isokinetic conditions has no significant effect on measured particulate concentration. The error in measured particle content as a function of an isokinetic velocity mismatch can be estimated analytically (see Figure 5-1). A comparison of duct and sampling velocities and the calculated correction factors for anisokinetic conditions is presented in Table 5-4. As shown, the measured particulate concentrations are within 1 or 2 percent of isokinetic measurements.

Table 5-3 lists the instrument readings from the gas sampling equipment used during Run 1. Gas sample flow was started shortly after particulate sampling began, so the total elapsed time is less than shown

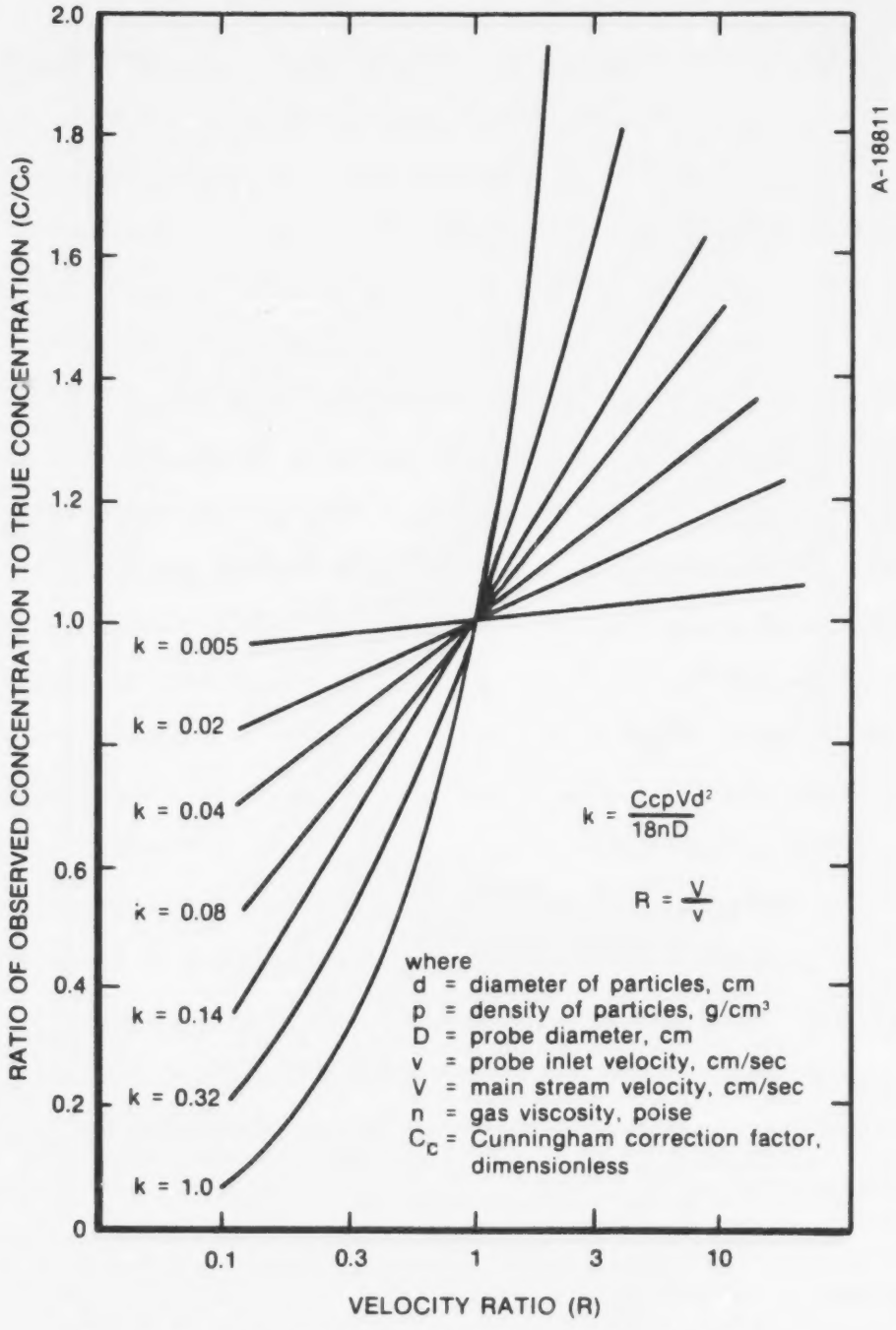


Figure 5-1. Probe inlet bias (from Reference 3).

in Table 5-2. During gas sampling, all sample flow was diverted to the gas train, so the flowrates given for the gas train are the same as those for the particulate collector. The temperature readings show that all gas train components were operating correctly.

During the test series, the surface temperatures of the access port, gate valves and probe housing were measured. These readings are presented in Table 5-5. The gate valve surface temperature remained below 126°C (258°F) at all times . Accessible surfaces of the probe housing also remained cool, below 75°C (167°F).

5.2 PHASE I -- DEMONSTRATION TEST RESULTS

The tests produced data on particulate concentration, size distribution, appearance, chemical composition, and moisture content. Although trace organic and trace element samples were collected, demonstrating that particular HTHP system capability, the analysis of these samples was beyond the scope of the program.

The measured particle concentrations are listed in Table 5-6. The values of 1.06 to 1.58 g/m^3 (0.43 to 0.64 gr/scf) are reasonable compared to other measurements made in the unpressurized portions of the Exxon process. Those measurements have ranged from 0.5 to 2.96 g/m^3 (0.2 to 1.2 gr/scf). The 1.58 g/m^3 (0.64 g/scf) value from the 30-minute sample is the most accurate measurement from the current tests. It comes from the largest sample and best defined conditions.

The moisture content measured in Run 1 was 6.2 percent by volume. This compares well with Exxon's preliminary estimate of 5.8 percent.

Particle size distribution information is presented in Figure 5-2 and Table 5-7. As shown, there is some difference in the results from

TABLE 5-5. STRUCTURE TEMPERATURES

Pretest (11:00 a.m., 3-30-77)

Duct Wall	176°C (349°F)
Pressure Cylinder	
Top	146°C (294°F)
Side	138°C (280°F)
Bottom	87°C (189°F)
Gate Valve - Duct Side	
Top	126°C (258°F)
Side	104°C (220°F)
Bottom	91°C (195°F)

Run No. 1 (3:30 p.m., 3-31-77)

Time	Valve-Probe Side	Inner Probe Housing	Outer Probe Housing
0	64°C(147°F)	56°C(133°F)	56°C(133°F)
10 min	71°C(159°F)	55°C(131°F)	68°C(154°F)
20 min	69°C(157°F)	56°C(133°F)	75°C(167°F)
60 min	88°C(190°F)	N/A	N/A

TABLE 5-6. PARTICULATE CONTENT

	Run #1	Run #2	Run #3
Date:	3-31-77	4-1-77	4-1-77
Time:	1530	1030	1500
Particle Catch: (grams)			
Filter	3.2515	-	-
Impactor	-	0.0554	0.0892
Residue	1.8565	0.0334	0.0595
Total	5.108	0.0884	0.1497
Sample Volume: m^3(scf)	3.22(122.5)	0.082(3.13)	0.132(5.03)
Particle Content: (g/m^3)(gr/scf)	1.58(0.64)	1.06(0.43)	1.13(0.46)
Particle content: (g/m^3)(gr/scf) (Anisokinetic Correction Applied)	1.60(0.65)	1.08(0.44)	1.16(0.47)

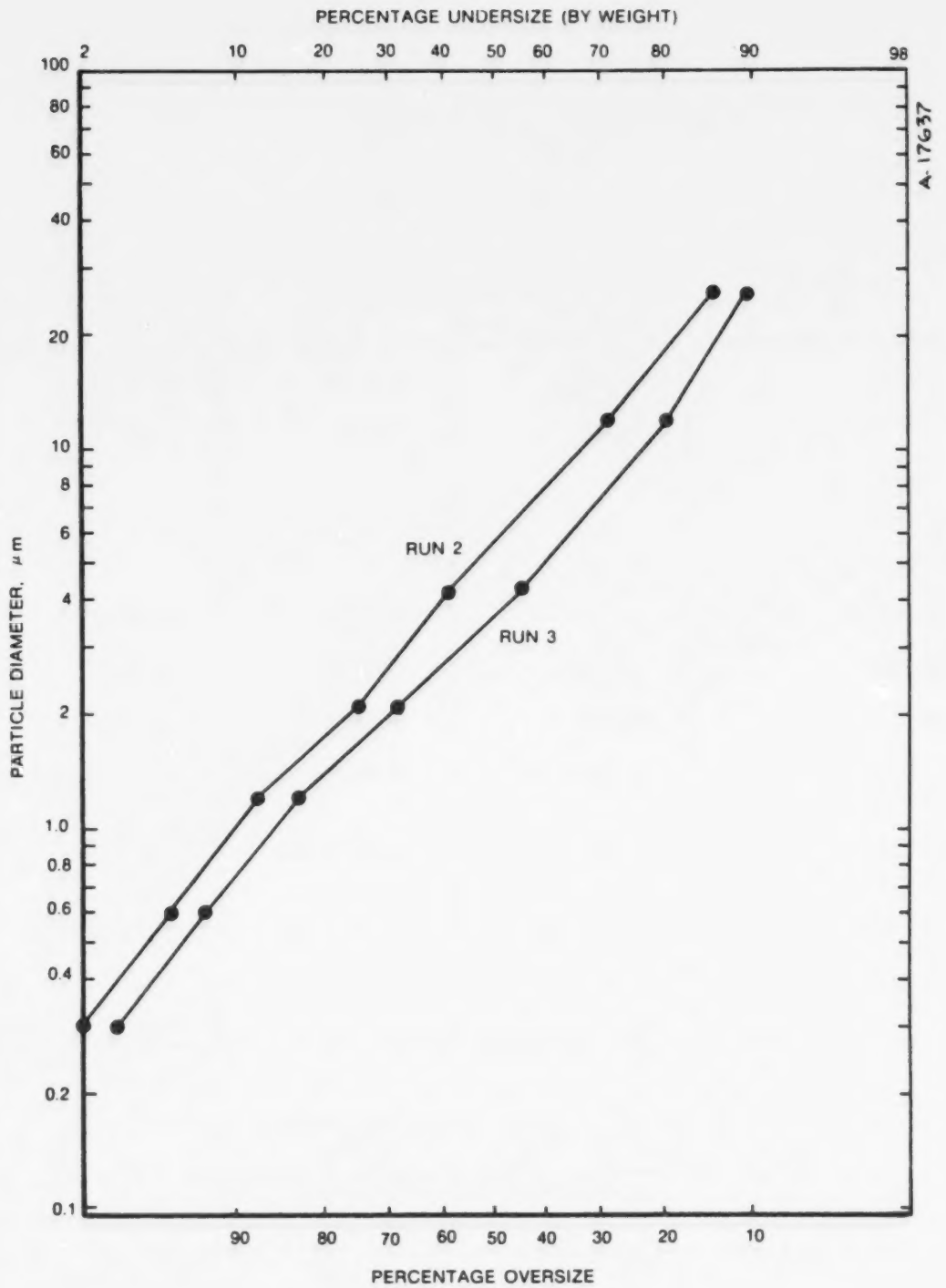


Figure 5-2. Particle size distribution -- Phase I tests.

TABLE 5-7. PARTICLE SIZE DISTRIBUTION

Stage	D_{50} Microns	Run #2			Run #3		
		Weight Collected	% Total Weight	% Smaller	Weight Collected	% Total Weight	% Smaller
1	26.0	0.0076	13.7	86.3	0.0093	10.4	89.6
2	12.0	0.0080	14.4	71.8	0.008	9.0	80.6
3	4.3	0.0171	30.9	41.0	0.0221	24.8	55.0
4	2.1	0.0139	25.1	15.9	0.0215	24.1	31.7
5	1.2	0.0022	4.0	12.0	0.0135	15.1	16.6
6	0.6	0.0036	6.5	5.4	0.0081	9.1	7.5
7	0.3	0.0020	3.6	1.8	0.0039	4.4	3.1
Filter		0.0010	1.8		0.0028	3.1	
		0.0554 grams			0.0892 grams		

the two impactor runs. However, both show that most of the particulate falls within the 1-to 20-micrometer range.

The impactor substrates are shown in Figures 5-3 through 5-6. Generally, the patterns are typical of normal impactor operation. Stage 7, however, shows evidence of several plugged jets. A comparison of Figures 5-4 and 5-5 show the differences in particulate loading for substrates from Run 2 and Run 3. Run 2 substrates were lightly loaded, while those from the longer duration Run 3 showed heavy, three-dimensional deposits.

The particulate sample from Run 2 were photographed using a scanning electron microscope (see Figures 5-7 through 5-10). The particulate is irregular in appearance, suggesting that it may be calcium sulphate crystals from the dolomite bed and ash from low-temperature combustion. Some of the photos show congealed masses of particles. The source of this phenomena could be any of the following: a property of the collected particulate, condensation on the particulate, or the conductive spray applied to the sample for SEM photography.

The chemical composition of collected particulate was analyzed by dispersive X-ray fluorescence analyzer. Spectra of X-ray emissions are shown in Figure 5-11. The peak heights for each element indicate the relative elemental concentration. The analysis shows detectable amounts of aluminum, silicon, calcium, sulphur, iron, potassium, titanium and copper.

5.3 PHASE II -- CONDENSATION TEST DATA

5.3.1 Test Conditions

Plant operating conditions are listed in Table 5-8. As in the Phase I tests, the nominal conditions were similar for all test runs. In

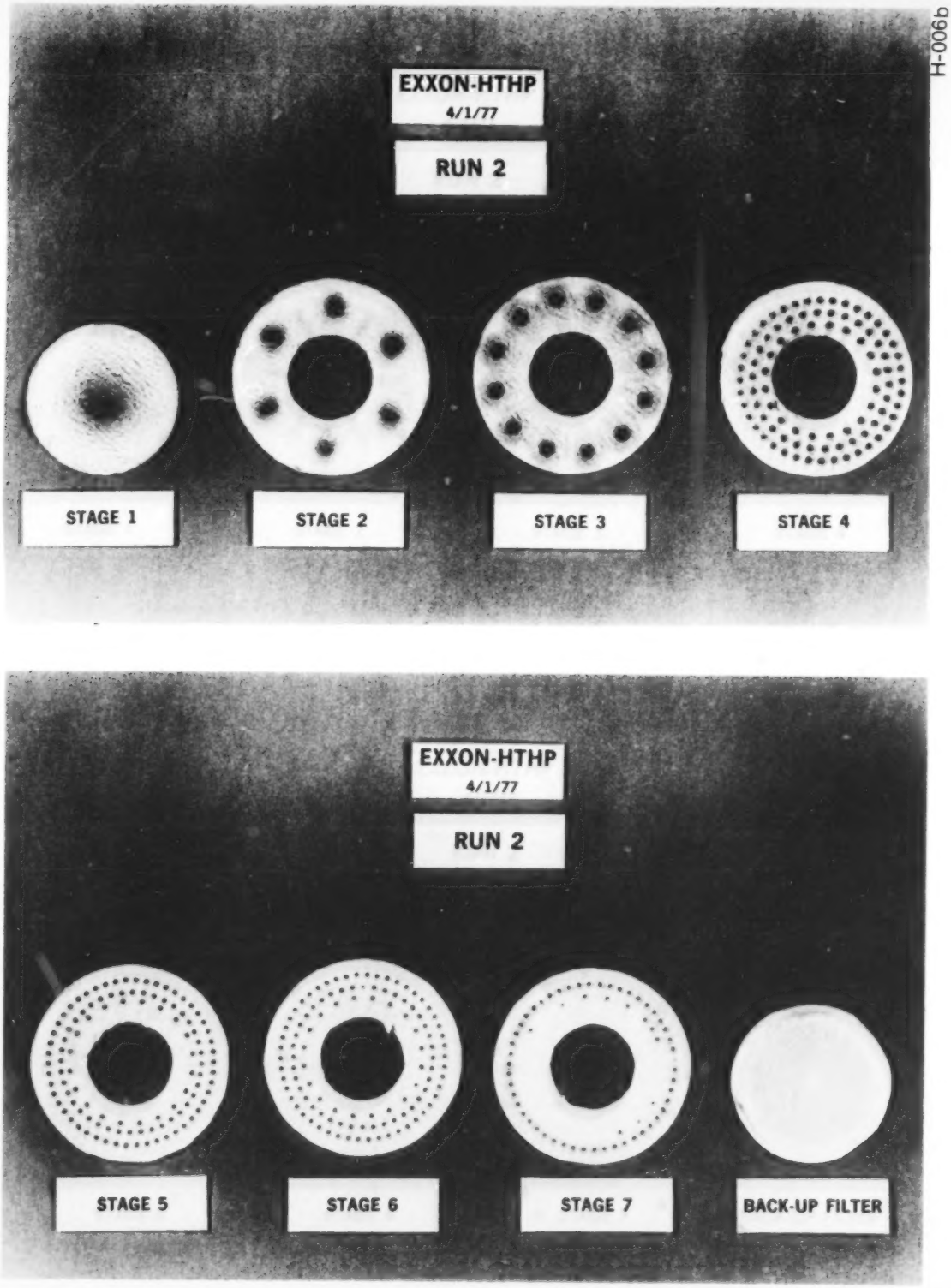
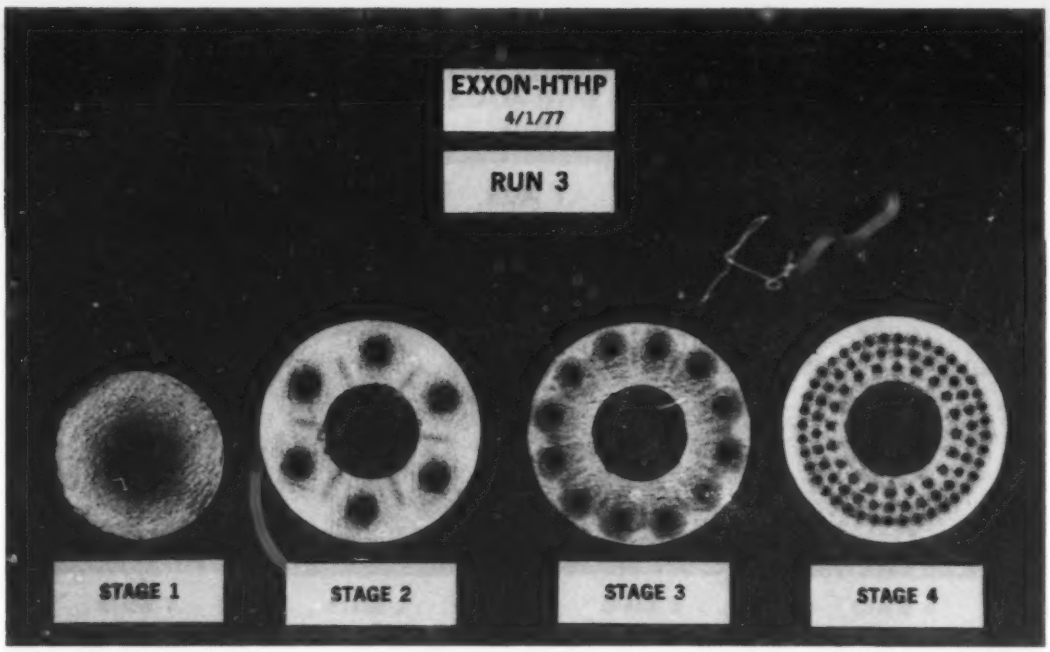
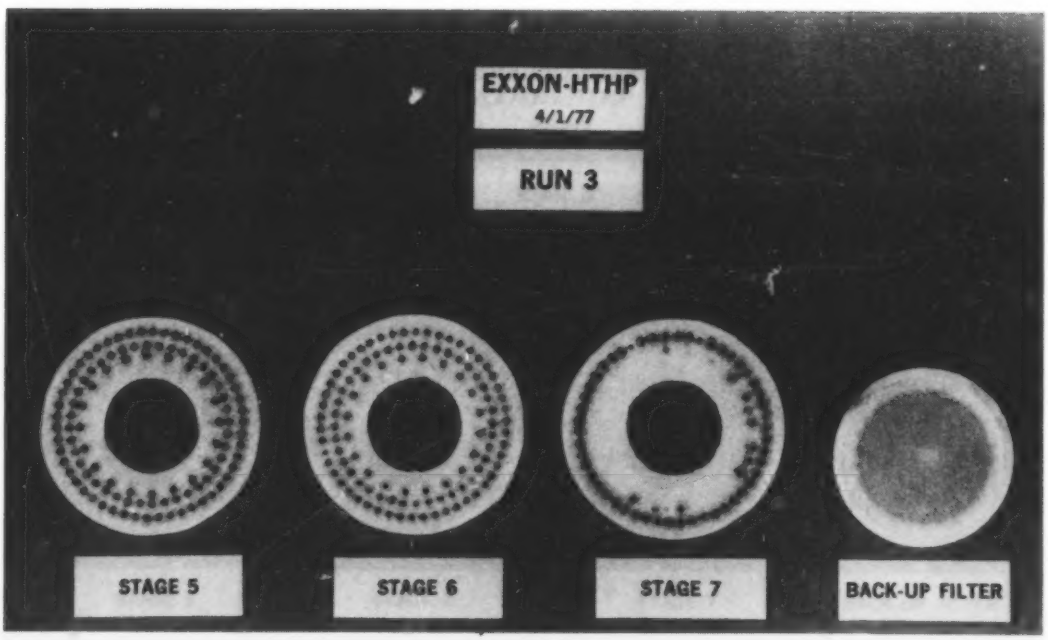

EXXON-HTHP
4/1/77
RUN 2
STAGE 1
STAGE 2
STAGE 3
STAGE 4
EXXON-HTHP
4/1/77
RUN 2
STAGE 5
STAGE 6
STAGE 7
BACK-UP FILTER
H-006b

Figure 5-3.



Impactor substrates.



Impactor substrates.

Figure 5-4.

Preceding page blank

H-0036

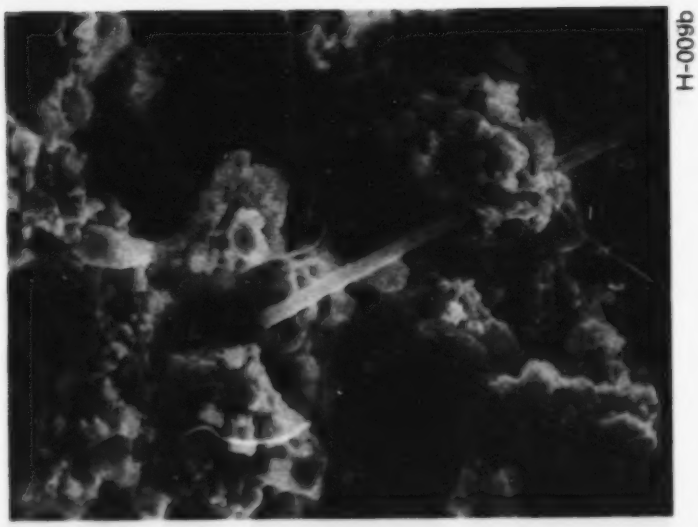
Figure 5-5.
Impactor substrate Run 3, Stage 5.

Preceding page blank

H-0046

Figure 5-6.
Impactor substrate – Run 2, Stage 5.

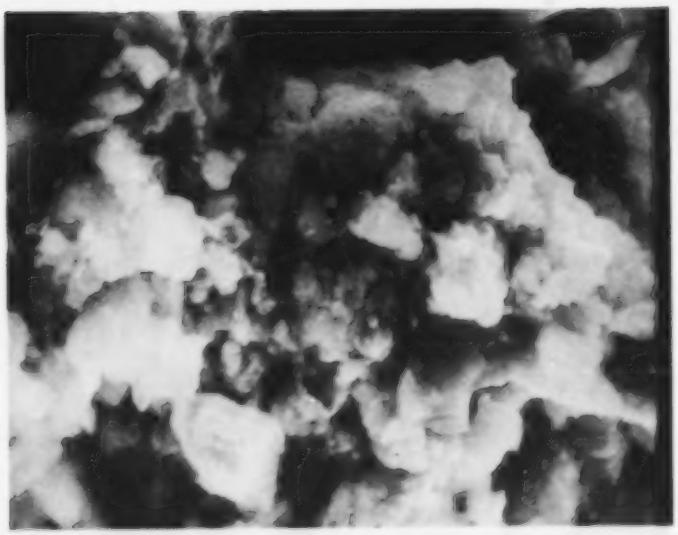
Preceding page blank



H-0696

1000X

⟷ 10 Microns



3000X

Figure 5-7.

Particle photomicrographs Stage 1.

Preceding page blank

1000 X

10 Microns

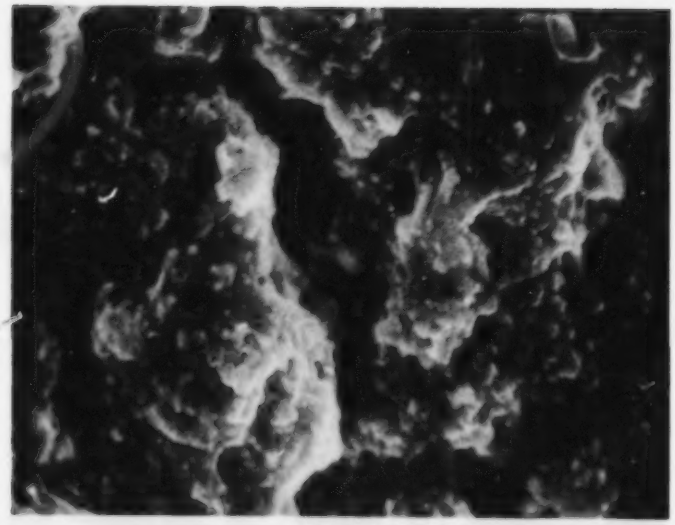
3000X

3 Microns

Figure 5-8.

Particle photomicrographs Stage 2.

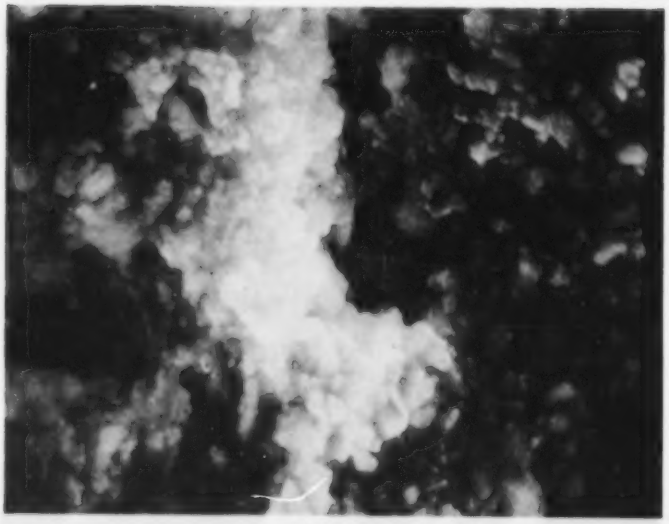
Preceding page blank



H-008b

1000X

10 microns

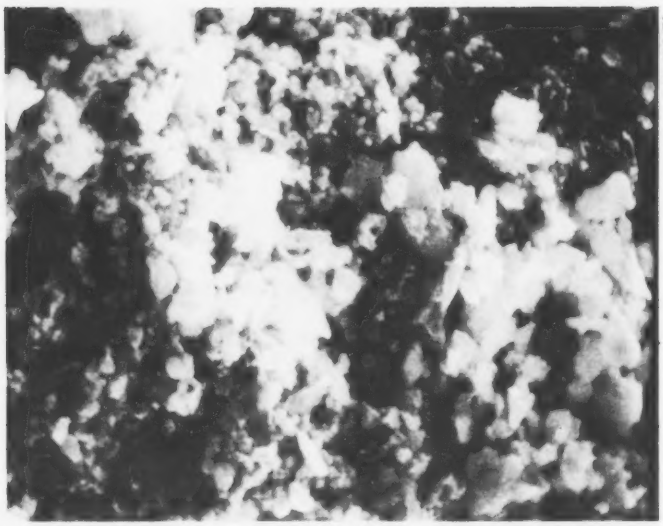


3000X

3 microns

Figure 5-9.
Particle photomicrographs Stage 4.

Preceding page blank



3000X

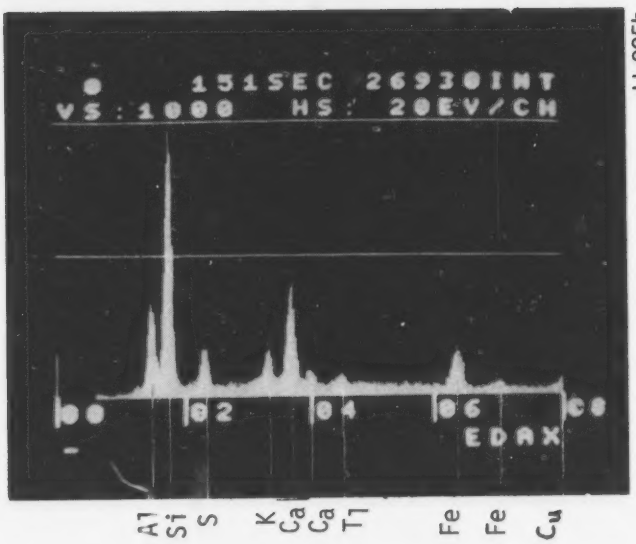
10000X

⟷ 1 micron

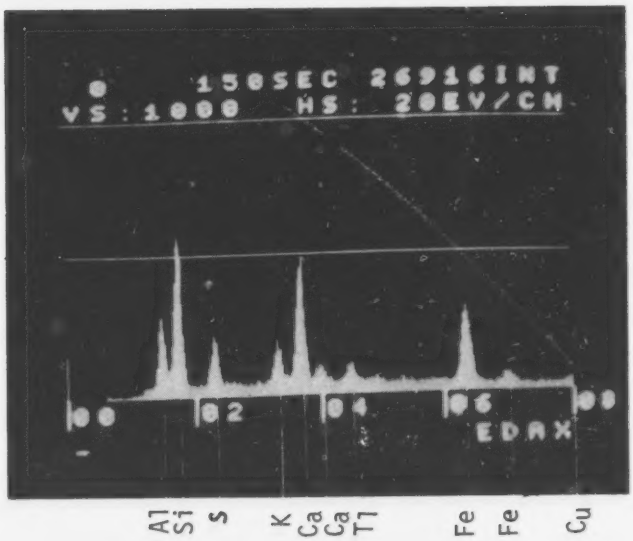
Figure 5-10.

Particle photomicrographs Stage 6.

Preceding page blank



Stage 1, Run 2



Stage 6, Run 2

Figure 5-11. Particle checmical composition.

Preceding page blank

Preceding page blank

TABLE 5-8. EXXON - PHASE II FBC OPERATING CONDITIONS

Run	1	2	3	4
Date	5-24-77	5-24-77	5-24-77	5-24-77
Sampling Time	1433 - 1503	0900 - 0920	1134 - 1153	1700 - 1715
Ambient Temperature °C(°F)	32(90)	29(85)	29(85)	31(88)
Bed Conditions				
Temperature °C(°F)	893(1640)	891(1635)	893(1640)	896(1644)
Combustor Pressure kPa(atm)	932.2	932.2	922.1	922.1
Ca/Sulphur Ratio	1.25	1.25	1.25	1.25
Coal	CHAMPION	CHAMPION	CHAMPION	CHAMPION
Dolomite	PFZIZER	PFZIZER	PFZIZER	PFZIZER
Combustor Flowrate - sm^3/min	16.5(628)	16.6(630)	16.6(631)	16.8(639)
Avg. Duct Velocity - m/sec(ft/sec)	2.43(7.97)	2.44(7.99)	2.44(800)	2.47(8.11)

fact, the test conditions for both phases of testing were very similar. Champion coal from the same shipment and Pfzizer dolomite were used for all testing. Combustor flowrate and average duct gas velocity were approximately 15 percent greater in the Phase II tests. The facility ran steadily with no interruptions during the tests.

5.3.2 Instrument Readings

The Phase II sampling system employed the same instrumentation control modules as the Phase I tests. Readings of duct, sample and equipment operating conditions are presented in Table 5-9.

The stack gas thermocouple malfunctioned throughout the four tests. This temperature was approximated at 732°C (1350°F) -- the average stack temperature during the first test series. Very similar PFBC operating conditions between the two test series make this a reasonable approximation. Another less critical malfunction occurred in the transport tube outlet filter thermocouple. This problem had the nature of an intermittent electrical short. Consequently, the measurement could be made only periodically.

The sample conditioning data showed the sample temperature falling below the target of 232°C (450°F). Temperatures sometimes fell as low as 93°C (200°F) immediately after the flow control valve and rarely exceeded 204°C (400°F) in the transport tube leading to the final filter. Assuming that flue gas SO_3 concentrations are below 30 ppm, as they are in conventional fossil fuel fired boilers, then Figure 5-12 indicates the H_2SO_4 dewpoint temperature to be between 177°C (350°F) and 204°C (400°F). Consequently, it should be expected that formation of sulfuric acid mist occurred in the transport tube. Upon analysis of the rear filter catch, this was found to be the

TABLE 5-9. PROBE INSTRUMENTATION READINGS

	Time of Day	Elasped Time (Min)	Stack Pressure kPa(psi)	Stack Temp °C(°F)	Dowtherm Inlet Temp °C(°F)	Dowtherm Outlet Temp °C(°F)	Traverse Tube Heater Temp 2 °C(°F)	Traverse Tube Outlet Temp 3 °C(°F)	Traverse Tube Outlet Filter Temp 4 °C(°F)	Sample Flowrate $m^3/sec \times 10^{-4}$ (acfm)	Transport Tube Temp °C(°F)
Run No. 1											
5-24-77 Tues. Zero Point	1333	0	882.5(128)	M	185(365)	188(370)	206(403)	41(106)	33(91)	2.41(.51)	277(530)
	1351	5	765.3(111)	M	188(370)	193(380)	201(394)	102(215)	34(93)	2.41(.51)	277(530)
	1358	10	758.4(110)	-	182(360)	188(370)	203(397)	122(252)	33(92)	2.41(.51)	277(530)
	1404	15	758.4(110)	-	188(370)	193(380)	203(397)	125(257)	33(92)	2.41(.51)	277(530)
	1408	20	758.4(110)	M	189(372)	194(372)	202(395)	124(255)	34(93)	2.41(.51)	277(530)
	1412	25	758.4(110)	-	188(370)	193(380)	201(393)	129(265)	33(92)	2.41(.51)	277(530)
	1416	29	758.4(110)	-	189(372)	194(381)	202(395)	129(264)	34(93)	2.41(.51)	277(530)
Closed Ball Valve		30									
Retract Probe	1420	Stop-off	758.4(110)	-	188(370)	188(370)	202(395)	71(160)	33(92)	2.41(.51)	OFF
Run No. 2											
5-24-77 Tues. Zero Point		0	765.3(111)	510(950)	182(360)	188(370)	209(409)	36(97)	33(91)	2.41(.51)	293(560)
	2100	0	758.4(110)	493(920)	188(370)	193(380)	203(397)	141(285	163(325)	2.41(.51)	293(560)
	2105	5	758.4(110)	493(920)	185(365)	193(380)	203(398)	148(298)	173(344)	2.41(.51)	249(480)
	2110	10	765.3(111)	493(920)	187(368)	193(380)	204(400)	144(291)	174(345)	2.41(.51)	254(490)
	2115	15	765.3(111)	493(920	189(372)	194(381)	206(402)	144(291)	176(349)	2.41(.51)	257(495)
	2119	19	765.3(111)		189(372)	——	—— Closed	Value ——	——		257(495)
Closed Ball Valve		20									
Retract Probe	2122	Stop-off	765.3(111)		182(360)	188(370)	208(407)	86(187)	84(183)	2.41(.51)	OFF

Note: M indicates malfunction

TABLE 5-9. (Concluded)

	Time of Day	Elapsed Time (min)	Stack Pressure kPa (psi)	Stack Temp. °C (°F)	Dowtherm Inlet Temp. °C (°F)	Dowtherm Outlet Temp. °C (°F)	Traverse Tube Heater Temp. °C (°F)	Traverse Tube Inlet Temp. °C (°F)	Traverse Tube Outlet Filter Temp. °C (°F)	Sample Flow Rate $m^3/sec \times 10^{-4}$	Transport Tube Temp. °C (°F)
Run No. 3											
5/25/77, Wed.	11:12 a.m.	-5	834.3 (121)	M	188 (370)	188 (371)	211 (411)	36 (97)	34 (93)	2.41 (0.51)	266 (510)
Zero Point	11:18 a.m.	0	841.2 (122)	M	189 (372)	188 (370)	208 (407)	37 (99)	35 (95)	2.41 (0.51)	271 (520)
Prestart	11:34 a.m.	0'	765.3 (111)	M	188 (370)	188 (370)	211 (411)	38 (100)	37 (98)	2.41 (0151)	282 (540)
	11:41 a.m.	5	765.3 (111)	M	194 (382)	198 (389)	206 (405)	149 (300)	172 (341)	2.41 (0.51)	268 (515)
	11:46 a.m.	10	765.3 (111)	M	191 (375)	193 (379)	206 (403)	152 (305)	177 (351)	2.41 (0.51)	268 (515)
	11:51	15	765.3 (111)	M	188 (370)	188 (370)	206 (402	152 (305	179 (354)	2.41 (0.51)	268 (515)
Closed Ball Valve		19		M							
	11:58	Stopoff	765.3 (111)	M	177 (350)	182 (360)	87 (188)	79 (175)	--	2.41 (0.51)	Off
Run No. 4											
5/25/77, Wed.	4:55 p.m.	-5	834.3 (121)	M	191 (375)	189 (372)	206 (402)	41 (105)	35 (95)	2.41 (0.51)	263 (505)
	5:00 p.m.	0	765.3 (111)	M	196 (385)	199 (390)	199 (390)	135 (275)	158 (316)	2.41 (0.51)	271 (520)
	5:02 p.m.	5	765.3 (111)	M	192 (378)	193 (380)	201 (394)	151 (303)	171 (340)	2.41 (0.51)	266 (510)
	5:07 p.m.	10	765.3 (111)	M	193 (380)	194 (381)	202 (396)	148 (299)	173 (344)	2.41 (0.51)	266 (510)
	5:11 p.m.	14	765.3 (111)		196 (385)	197 (387)	201 (394)	142 (288)	172 (341)	2.41 (0.51)	271 (520)
Ball Valve Closed		15		Stopoff			System Down				
		01		M	179 (355)	182 (360)	Off	78 (173)		2.41 (0.51)	Off

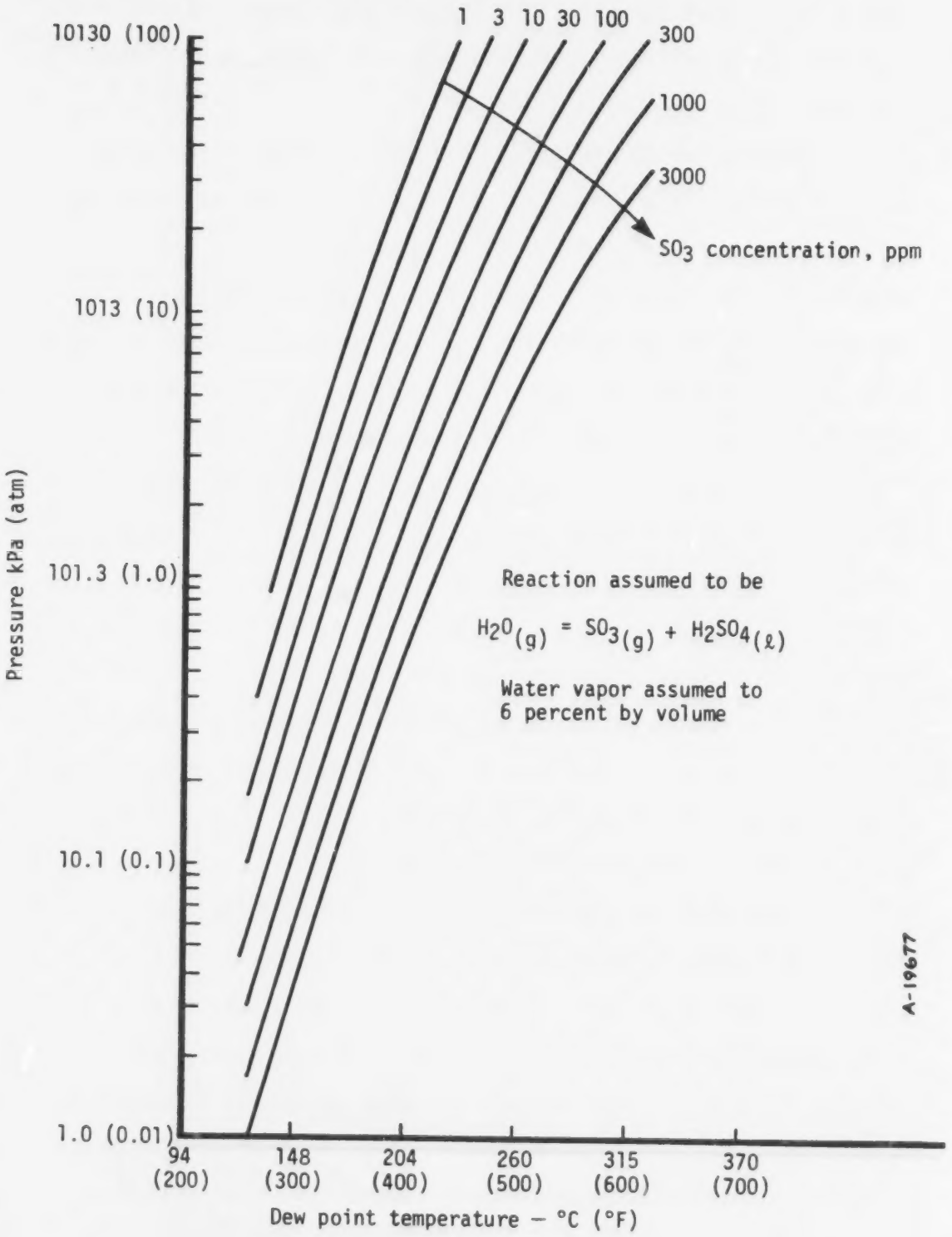


Figure 5-12. Condensation of H_2SO_4 (Reference 2).

case (see following section). This analysis also showed contamination from the disintegration of control valve packing located just upstream of the rear filter.

Sampling flowrates were predetermined by assuming a duct velocity, pressure and temperature, based on the Phase I tests, and appropriately sizing a nozzle and the choked orifices to yield isokinetic nozzle velocities. The actual sample flowrate gave nozzle velocities approximately 15 percent below duct velocities. However, as was the case with the Phase I tests, the high gas temperatures and pressures, the fine particles and low velocities involved minimized the effect of anisokinetic conditions on the measured particulate concentration. Furthermore, since the purpose of the Phase II tests was to examine only elemental concentrations in the hot and cold filters, particle size biasing caused by anisokinetic sampling conditions was relatively unimportant. A comparison of duct and sampling velocities and the calculated correction factors for anisokinetic conditions is presented in Table 5-10. The measured particulate concentrations are within 3 percent of the true concentration.

5.4 PHASE II -- CONDENSATION TEST RESULTS

The condensation test results section is divided into three subsections. General results and observations of the samples, both visually and by photomicrographs, are discussed first. Attempts at comparing the analyses of the hot and cold filter catches and the associated problems are treated next. Finally, an alternate approach to answering the question of condensation of trace elements is discussed. Tentative conclusions are drawn from the limited data obtained.

TABLE 5-10. ANISOKINETIC CORRECTION FACTORS - PHASE II

Run	Sample Flowrate $m^3/s \times 10^{-4}$ (acfm)	Nozzle Velocity m/sec (ft/sec)	Estimated Duct Velocity m/sec (ft/sec)	Velocity Ratio	Particle Concentration[a] Correction Factor $\frac{C_{measured}}{C_{true}}$
1	2.4(.51)	2.2(7.1)	2.4(8.0)	1.13	1.03
2	2.4(.51)	2.2(7.1)	2.4(8.0)	1.13	1.03
3	2.4(.51)	2.2(7.1)	2.4(8.0)	1.13	1.03
4	2.4(.51)	2.2(7.1)	2.4(8.1)	1.14	1.03

[a]Calculation per method in Handbook of Aerosols, TID-26608, 1976, Section 5.1-1 and Figure 5-2.

5.4.1 General Results and Observations

Table 5-11 shows particulate concentration. As noted, Run No. 3 samples were retained by EPA for SSMS analysis and, consequently, not weighed. The reported values for particulate catch have been obtained by using standard EPA drying and weighing techniques. The values of 0.99 to 2.49 g/m^3 (0.40 to 1.01 gr/scf) are reasonable compared to other measurements made on the FBC. As stated earlier, these values have ranged from 0.49 to 2.96 g/m^3 (0.2 to 1.2 gr/scf) on a wet basis. A flue gas moisture content of 6 percent could be assumed if it is desired to obtain dry basis particulate concentrations. This is an approximate value from the Phase I test series.

Visual observations of the front and rear filters from Tests 1, 2, and 4 are summarized in Table 5-12. In addition, all these samples were photographed using a scanning electron microscope.

As in the first test series, the particulate is irregular in appearance, suggesting that it may be calcium sulfate crystals from the dolomite bed and ash from low-temperature combustion. However, some of this particulate is undoubtedly gold deposited as a result of corrosion, blistering, and flaking of the gold plating on the scalping cyclone. Chemical analysis of the hot filter catch showed considerable gold concentrations -- many times greater than would be expected in flyash. This gold contamination has little negative effect on the condensation test results. The photographs in Figures 5-13 and 5-14 show agglomerated particles. For the front filter, the cause could be either a property of the collected particulate or the conductive spray applied to the sample

TABLE 5-11. PARTICULATE CONCENTRATION

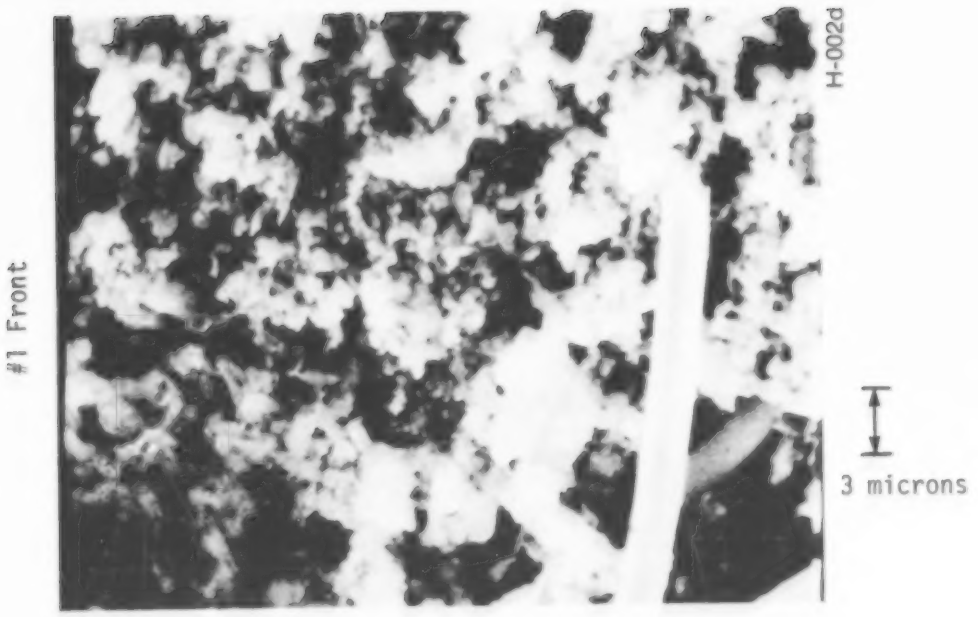
	Run #1	Run #2	Run #4
Date	5-24-77	5-24-77	5-25-77
Time	1445	0910	1710
Particulate Catch (grams)			
Cyclone	1.414	2.354	1.243
Front Filter	.374	.654	.598
Rear Filter	.014	.014	.006
Probe Wash	.266	.469	.359
TOTAL	2.068	3.491	2.206
Sample Volume Nm^3(scf)	2.05(78)	1.37(52)	1.03(39)
Particle Content g/m^3/(gr/scf)	1.01(.41)	2.56(1.04)	2.14(.87)
Particle Content g/m^3/(gr/scf) (Anisokinetic Correction Applied)	.99(.40)	2.49(1.01)	2.07(.84)

Note: Run 3 samples retained by EPA for SSMS analysis - no weights available.

TABLE 5-12. VISUAL OBSERVATION OF FILTERS — PHASE II

Test	Front (all disintegrating)	Rear
1	Flesh color - fine particles light loading	Dark tan - very few metallic-like particles, medium coating of fine particles
2	Flesh color - fine particles medium loading	Dark tan - more metallic-like particles, medium coating of fine particles
4	Flesh color - fine particles medium loading	Grey with a tan tinge - many metallic-like particles, light coating of fine particles, 4- to 5-mm hole either abraded or corroded in center of filter

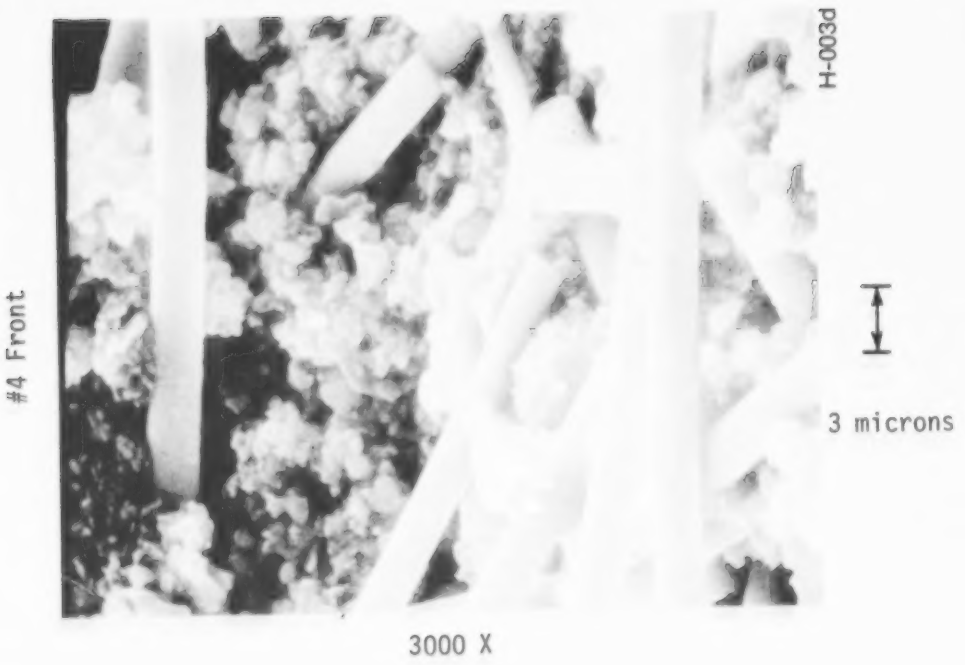
Note: No observations made on Test 3 — filters given to EPA for SMS analysis.



3000 X

10000 X

Figure 5-13. Particle photomicrographs -- Run 1, front filter.



3000 X

10000 X

Figure 5-14. Particle photomicrographs -- Rund 4, front letter.

Preceding page blank

for SEM photography. For the rear filters, shown in Figures 5-15 and 5-16, these causes are again possibilities, in addition to the known existence of H_2SO_4 condensation and valve packing contamination.

The rear filter for Test 4, shown in Figure 5-15, is noticeably lighter in particulate than Tests 1 and 2 rear filters (Figure 5-13). This is because two front filters were used in Test 4 while only one was used in Tests 1 and 2. Figure 5-17 shows a photograph and a dispersive X-ray analysis of a blank rear filter. Dispersive X-ray fluorescence analysis for the Test 4 front filter (Figure 5-18) shows detectable amounts of aluminum, silicon, sulfur, potassium, calcium, titanium, and iron. The rear filter analysis from the same test shows that the compounds of most elements almost completely filtered out in the front filter.

The cyclone used was a Southern Research Institute model designed for much less severe operating temperatures. It was readily available, was small enough for insertion into the duct, and had a very efficient 0.6 micron cutpoint. As previously mentioned, the cyclone's protective gold plating blistered and peeled, leaving titanium surfaces exposed to heavy oxidation. Chemical analysis of the particulate samples showed significant gold contamination in the cyclone and front filter catches but none in the rear filter catch. Titanium contamination was not evident in any of the samples.

5.4.2 Initial Analyses for Condensation Effects

The original intention of the Phase II tests was to compare the elemental concentrations found in the filtered material of the in-stack scalping cyclone and backup filter with the material caught on the rear filter. If the system worked ideally, one could assume all the material

Preceding page blank

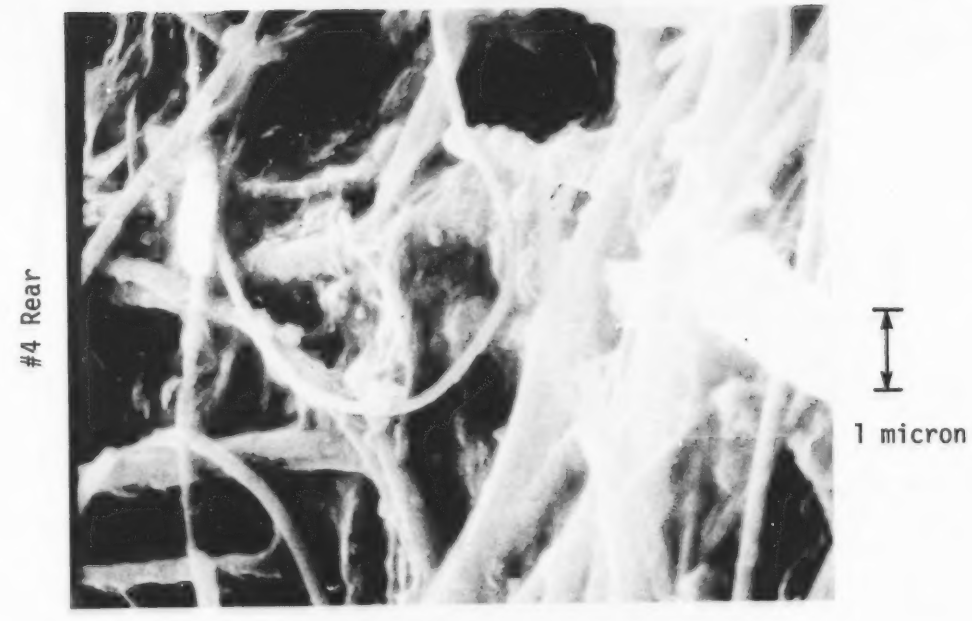
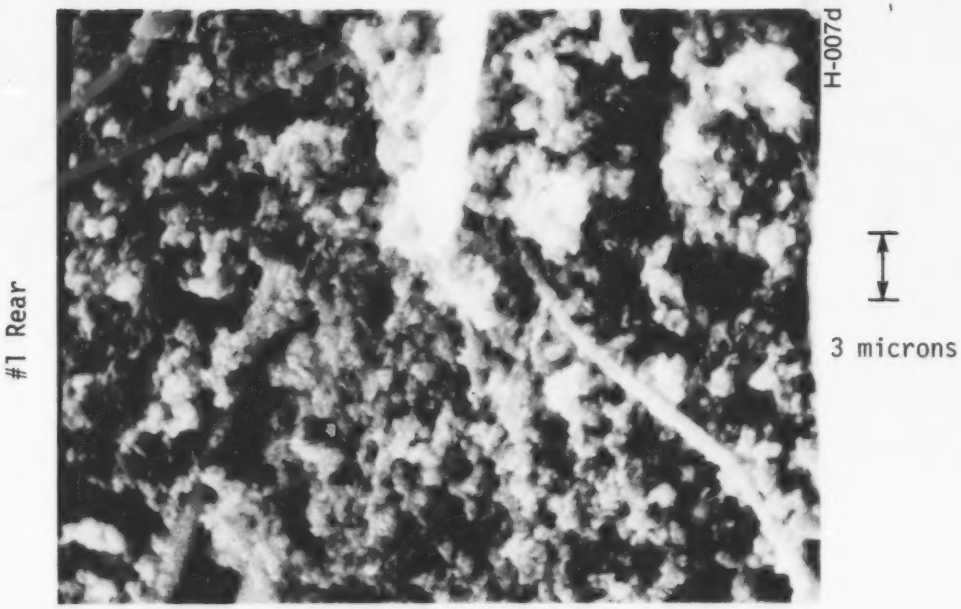
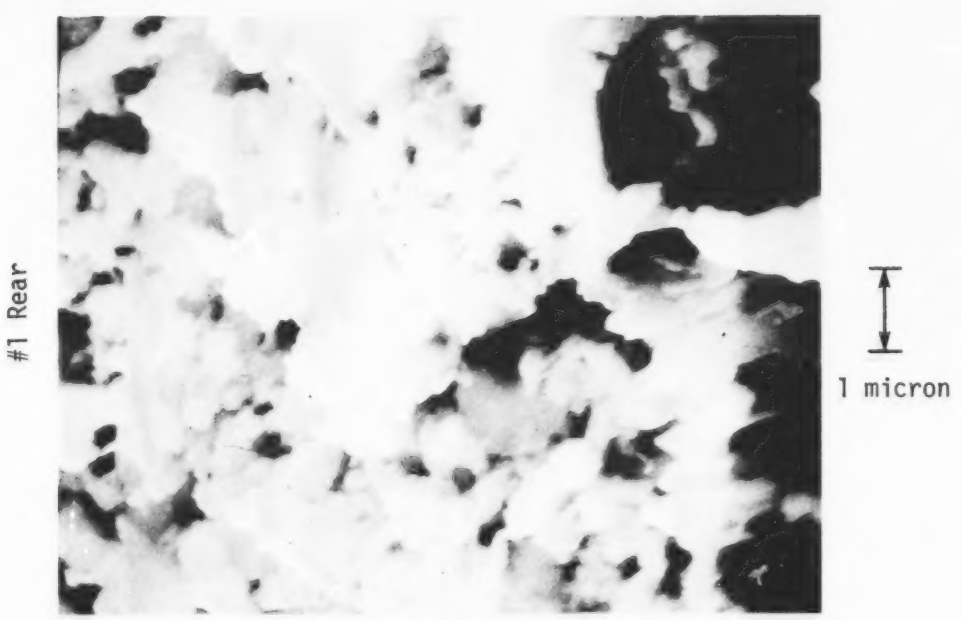


Figure 5-15. Particle photomicrographs -- Run 4, rear filter.

Preceding page blank



3000 X

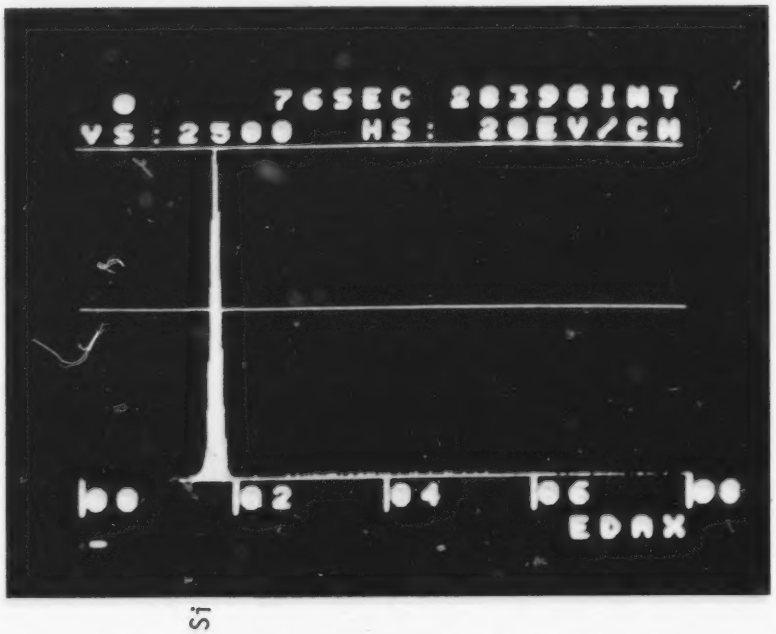


10000 X

Figure 5-16. Particle photomicrographs -- Run 1, rear filter.

10000 X

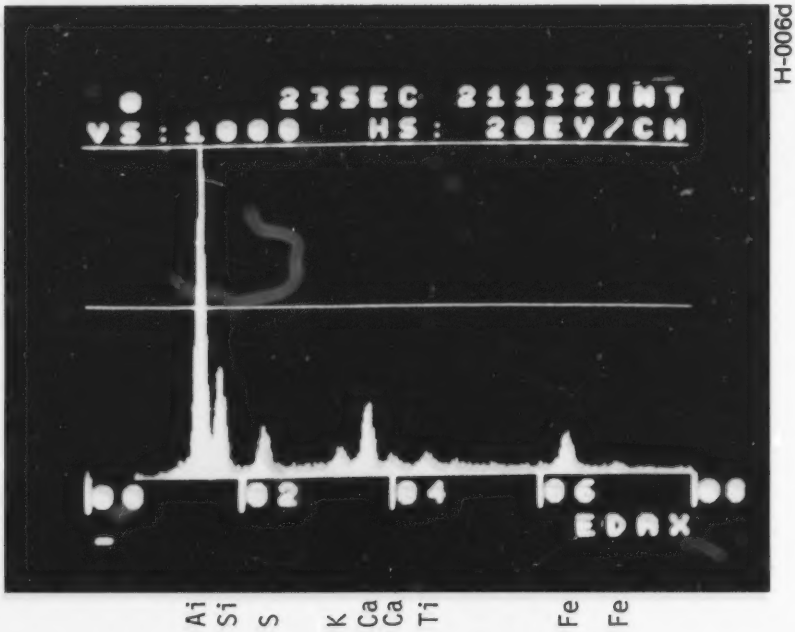
Particle photomicrograph — blank rear filter.



Particle chemical composition — blank rear filter.

Figure 5-17.

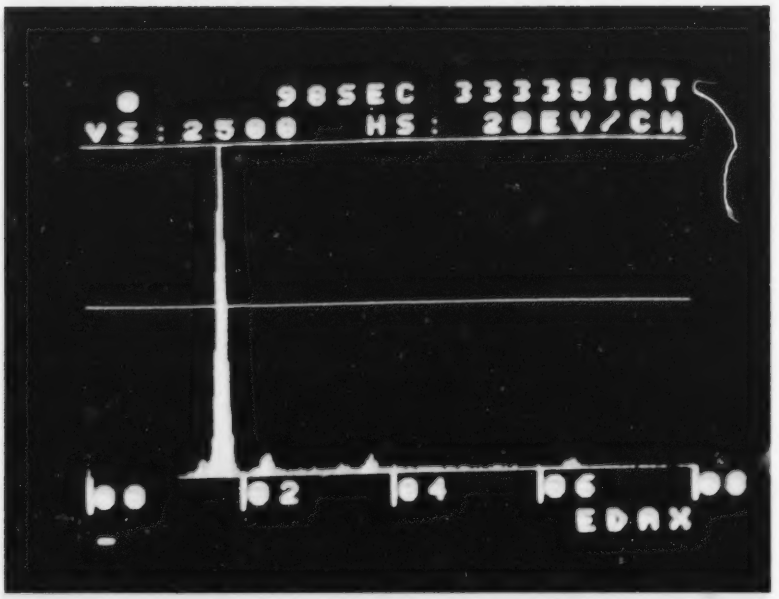
Preceding page blank



#4 Front

Al Si S K Ca Ca Ti Fe Fe

Front filter



#4 Rear

Al Si S K Ca Fe

Rear filter

Figure 5-18. Particle chemical composition - Run 4.

Preceding page blank

on the rear filter was in a gaseous state at stream conditions and would thereby pass through the hot cyclone/filter combination and thus be indicative of condensation products produced within the probe. Unfortunately, the rear (or cold) filter could not be reliably analyzed. Carryover of glass fiber filter material during sample preparation (the small amount of the sample available on the cold filter and additional contamination already mentioned), yielded poor detection limits with the spark source mass spectrometer (SSMS). Moreover, the values reported for this filter were in mass units rather than concentration units because a net filter collection weight was not determined. This made any direct comparisons of cold and hot catches from Phase II results alone difficult if not speculative.

Samples of the probe wash were analyzed by Arthur D. Little, Inc., to determine the source of the suspected contamination of the rear filter. A sample from each test was analyzed by thermal gravimetric analysis (TGA), infrared analysis (IR), X-ray fluorescence (XRF), and low resolution mass spectra (LRMS). Results indicated there were three sources of contamination. They were by weight: (1) approximately 30 percent particulate -- attributed to hot, in-stack filter breakthrough, (2) approximately 25 percent sulfuric acid and sulfate condensate -- attributed to localized cold spots (measured at 200°F, below the H_2SO_4 condensation temperature), and (3) approximately 40 percent organics -- later attributed to the disintegration of packing material from a valve located just upstream of the rear filter. The evidence of breakthrough particulate contamination was supported by photomicrographs, which showed similar appearance between material on the front and rear filters, and by dispersive fluorescent X-ray spectrum which shows a

Preceding page blank

similarity in chemical composition. The sulfur content did, however, increase on the rear filter, apparently as a result of sulfate condensation.

5.4.3 Alternate Approach

An alternate approach was taken to resolve the question of trace metal condensables. During the Phase I demonstration tests at Exxon, a test run was made at essentially the same operating and stream conditions as the Phase II tests. A total mass filter was used in place of the cascade impactor and was maintained at about the same temperature as for Phase II. Northrop Services, Inc. performed a SSMS analysis of the Phase I bulk filter catch, as they did with the Phase II hot cyclone/filter samples.

Table 5-13 presents the results for these analyses. The measured elemental content is similar to common flyash. A partial, nondimensionalized comparison of these two sets of results, Phase I and Phase II, is presented in Table 5-14. Reference quantities Fe and Mg, have been chosen to nondimensionalize the results because they exist in significant concentrations in each sample and are not likely to be present as a result of contamination. Nondimensionalizing was done because there appeared to be a diluent present in the Phase I sample filter catch. The Si concentrations indicate that the filter material itself may be the diluent in the sample. In fact, the sample analyst acknowledged some difficulty with separating the sample from the filtering media prior to analysis.

Aside from the silicon results, comparison of the other quantities shown indicates that there was no detectable change in the concentration of Na or K from the hot to cold particulate catches. It should be

TABLE 5-13. CONCENTRATION OF ELEMENTS IN FLYASH
SSMS ANALYSIS (PARTIAL)

Element	TEST #3 - PHASE II				PHASE I
	Cyclone (ppm)	Front (ppm)	Rear (ppm)	Rear Blank (ppm)	Bulk Filter (ppm)
K	8,200	8,850	15.1	5.4	16,250
Na	1,310	2,500	< 135. *	91.	3,560
Rb	< 70	< 68	< .43 *	.13	866
Cs	6.7	.23	< .62 *	------	8.8
Al	164,000	94,000	64.2	6.4	Major
Si	94,000	82,600	<2510. *	1210.	310,000
Fe	30,000	13,400	60.	2.36	36,300
Ca	20,000	19,000	< 6.6 *	7.3	44,000
Mg	11,400	17,800	38.	5.9	28,600
Ti	2,430	1,950	7.1	3.1	10,600
Sr	810	555	.6	1.15	1,320
Ba	710	694	< 5.0	1.5	1,080
Au	650	118	< 1.4	------	213
P	276	223	< 224. *	68.	1,880
Cu	248	165	< 1.5 *	.91	142
Zr	160	140	< 2.1 *	.64	334
Ni	120	100	5.8	.29	348
Cr	< 90	< 140	< 13.1	6.4	366
Pb	85	75	< 4.0 *	1.2	86

Notes: < - natural background limited detection limit
<* - blank limited detection limit

TABLE 5-14. PARTIAL COMPARISON OF FRONT AND REAR PARTICULATE CATCHES FROM EXXON TEST SERIES I AND II

	Phase I Tests Bulk Filter	Phase II Tests Cyclone	Front Filter	Avg.	Ratio
$\frac{K}{Fe}$	0.45	0.27	0.66	0.47	0.96
$\frac{Na}{Fe}$	0.10	0.04	0.19	0.12	0.83
$\frac{Si}{Fe}$	8.54	3.13	6.16	4.65	1.84
$\frac{Ca}{Fe}$	1.23	0.67	1.42	1.05	1.17
$\frac{Mg}{Fe}$	0.79	0.38	1.33	0.86	0.92
$\frac{Sr}{Fe}$	0.04	0.03	0.04	0.04	1.00
$\frac{Ba}{Fe}$	0.03	0.02	0.05	0.04	0.75
$\frac{K}{Mg}$	0.57	0.72	0.50	0.61	0.93
$\frac{Na}{Mg}$	0.12	0.11	0.14	0.13	0.92
$\frac{Si}{Mg}$	10.84	8.25	4.64	6.45	1.68
$\frac{Fe}{Mg}$	1.27	2.63	0.75	1.69	0.75
$\frac{Ca}{Mg}$	1.56	1.75	1.07	1.41	1.11
$\frac{Sr}{Mg}$	0.05	0.07	0.03	0.05	1.00
$\frac{Ba}{Mg}$	0.04	0.06	0.04	0.05	0.80

stressed that these are very limited data from two tests taken at different times comparing hot and cold filter catches. However, the PFBC was using the same coal and dolomite under very similar operating conditions for both tests, thereby supporting the validity of the comparison. Further testing is recommended to provide data points at other PFBC operating levels and possibly with different filtering media and collection temperatures.

SECTION 6

CONCLUSIONS

The sampling system described in this report demonstrates that extractive sampling is a feasible approach for sampling high-temperature, high-pressure processes. In addition, the limited data obtained at one PFBC operating level during the Phase II condensation tests, seems to indicate that trace element condensation would have minimal effect on particulate composition collected at a low temperature (approximately 400^{o}F). Technology for sampling pressurized fluidized bed combustors is now developed and available. Future development also will be required to make useful application of this technology and extend it to other advanced coal conversion processes.

One of the remaining issues for FBC high-temperature, high-pressure sampling is system cost/performance trade-offs. Process developers seem to be interested in both upgraded and downgraded versions of the sampling system. Upgraded versions offer longer sampling durations, quicker turnaround and better operating convenience. Downgraded versions, such as fixed-probe designs, are cheaper, but give less information.

The next objective for extractive sampling is to develop sampling technology for gasifiers. Particulate measurement is also important for developing these processes, and environmental difficulties are even more severe than for PFBC's.

REFERENCES

1. Hamersma, J. W., et. al., "IERL-RTP Procedures Manual: Level 1 Environmental Assessment," EPA-600/2-76-160a, June 1976.

2. JANAF, "Thermochemical Tables," Second Edition, the Thermal Research Laboratory, Dow Chemical Company, Midland, Michigan, 1971.

3. Dennis, Richard, "Handbook on Aerosols," U.S. Energy Research and Development Administration, 1976.